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Enantiomeric Ratios of Chlordane-Related Compounds in Air near the Great Lakes

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Some important environmental contaminants are chiral; therefore, enantiomeric ratios (ER) of these compounds can provide useful insights into their environmental fates. In this study, permethylated α- and γ-cyclodextrin gas chromatographic columns combined with electron capture and negative ionization mass spectrometry were used to measure the ER values of chlordane and related compounds. These techniques provided the selectivity and sensitivity necessary for these low-level environmental determinations. Studies using racemic and enriched standards confirmed that this analytical system gave reproducible and accurate ER measurements. From August 1994 through September 1995, 48 air samples were taken near Lake Erie, five air samples were taken near Lake Michigan, and six air samples were taken near Lake Superior. These samples were analyzed to determine the spatial trends in ER values. While there were slight differences between the various sites, there were considerable differences between the compounds. The overall ER for *cis*-chlordane was 1.05 ± 0.02, which is close to racemic. The overall ER for *trans*-chlordane was 0.88 ± 0.02, which is significantly different than racemic and from the measured *cis*-chlordane value. This discrepancy suggests that *trans*- and *cis*-chlordane are metabolized differently in the environment. The overall ER of *exo*-heptachlor epoxide was 1.99 ± 0.04; this large deviation from racemic indicates that this compound is an enzymatic degradation product of heptachlor.

Introduction

Technical chlordane, a ubiquitous and persistent pesticide, was used in the U.S. from 1945 (*1*) until it was banned in 1988 (*2*). This mixture of more than 140 compounds was used for killing lawn and garden pests, for termite control, and for pest control on crops (*1, 2*). Chlordane's components and metabolites are acutely toxic (*1, 3, 4*), are suspected carcinogens (*5*), and possibly have estrogenic effects (*6*). *cis*- and *trans*-Chlordane and heptachlor epoxide, a metabolite of heptachlor, (see Figure 1 for structures) are chiral, and each enantiomer has different biological properties and environmental fates (*7*−*10*).

When manufactured and introduced into the environment, these compounds are racemic. As they move through the environment, biological degradation changes the enantiomeric ratio [ER, defined as the amount of the (+) enantiomer divided by the amount of the (−) enantiomer]. Physical processes such as volatilization, photolysis, and OH radical reactions do not change the ER (*5, 11*). Clearly, by measuring the ER, more knowledge can be obtained about the sources, fates, metabolism, and transport of these compounds.

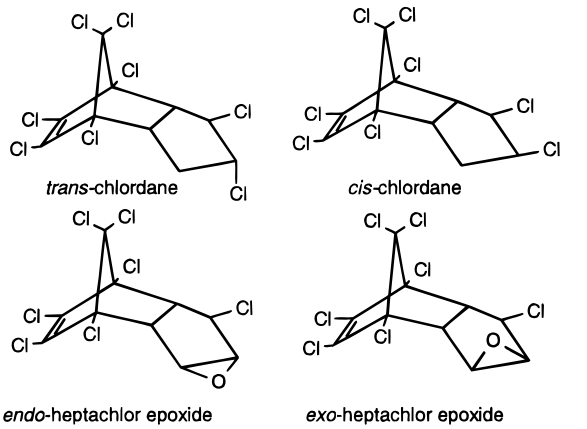

FIGURE 1. Structures of selected chlordane related compounds.

There have been several reports of chlordane ERs in aquatic biota (*10, 12*−*14*), in mammals (*9, 13*), in soils (*15*−*17*), and in water (*18*). With two exceptions, there have been no reports of chlordane ERs in air. The two exceptions are studies by Buser and Müller (*5*) and by Wiberg et al. (*15*), who reported ER values near 1.0 for heptachlor, *cis*- and *trans*-chlordane in Norwegian air and in southern U.S. air, respectively. Wiberg et al. found nonracemic values for the same three compounds in Great Lakes air and nonracemic values for heptachlor epoxide in southern U.S. air (*15*).

The goal of this research has been to investigate the spatial trends of chlordane ERs in gas-phase air samples taken near the Great Lakes. These data have been used to estimate the degree of metabolism of the various chlordane-related compounds as a function of location. This project is an extension of the Integrated Atmospheric Deposition Network (IADN) project, which is currently monitoring deposition of organochlorine compounds to the Great Lakes (*19*).

Experimental Section

Racemic standards were purchased from Ultra Scientific (North Kingstown, RI). These compounds were combined into a mixture of *cis*- and *trans*-chlordane, heptachlor, and *endo*- and *exo*-heptachlor epoxides at 1.0 ng/μL in hexane. Enantiomerically enriched standards were purchased from Axact Standards (Commack, NY). A solution of the enriched enantiomers of the above compounds and enriched oxychlordane was diluted with hexane to a concentration of 0.1 ng/μL.

Two different GC columns, capable of chiral separations, were used in this study because of their different separation characteristics and for confirming results. Both α- and γ-cyclodextrin-120 (CD) columns were purchased from Supelco (Bellefonte, PA). Both of these columns had lengths of 30 m, internal diameters of 250 μm, and film thicknesses of 0.25 μm.

A GC temperature program was developed for each column. For the α-cyclodextrin column, the temperature program was 50 °C for 1 min, ramped to 130 °C at 5 °C/min, ramped to 155 °C at 0.2 °C/min, ramped to 230 °C at 20 °C/min, and held for an additional minute. The total run

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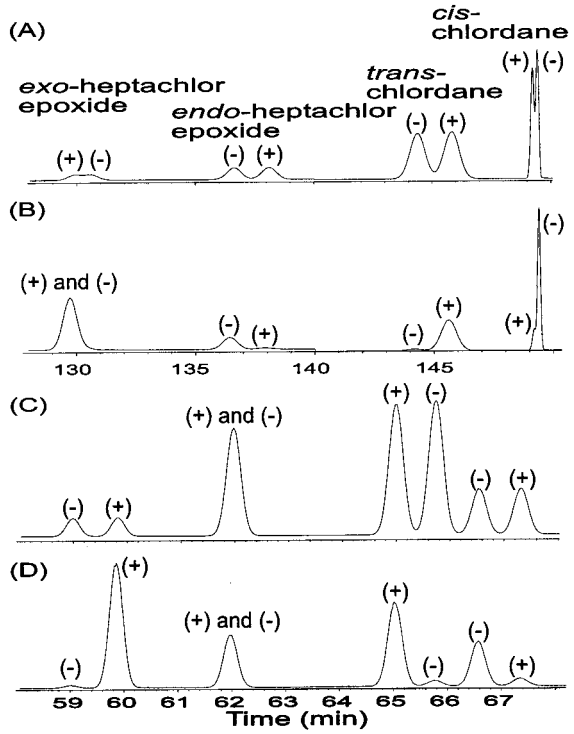

FIGURE 2. Chromatograms of (A) racemic mixture on α-cyclodextrin column, (B) enriched mixture on α-cyclodextrin column, (C) racemic mixture on γ-cyclodextrin column, and (D) enriched mixture on γ-cyclodextrin column. The narrow width of the *cis*-chlordane peaks in panels A and B is due to the faster temperature program rate that started at 142 min.

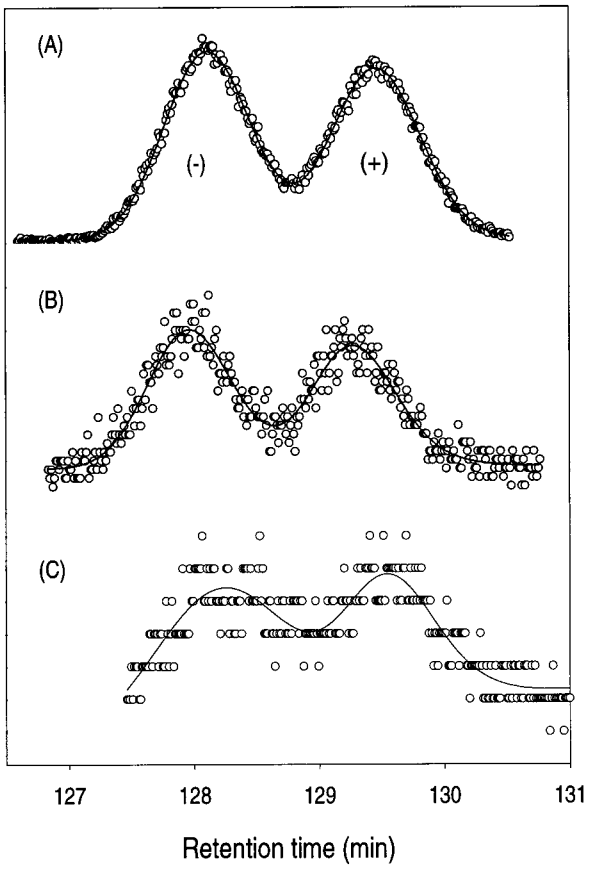

FIGURE 3. Portion of gas chromatogram showing *trans*-chlordane monitored at *m/z* 406 separated on a α-cyclodextrin column. (A) Good fit with a high *S/N*; concentration = 39 pg/m^3; ER = 0.92. (B) Acceptable fit with a lower *S/N*; concentration = 7.6 pg/m^3; ER = 0.86. (C) Unacceptable fit with a bad *S/N*; concentration = 0.5 pg/m^3; ER = 1.60.

time was about 150 min, and a constant carrier gas flow was used. The temperature program for the γ-cyclodextrin column also began with a 1 min hold at 50 °C, ramped to 150 °C at 20 °C/min, ramped to 185 °C at 0.5 °C/min, ramped to 230 °C at 20 °C/min and held for 1 min. The total run time was about 80 min, and a constant head pressure was used in this case. Both columns used a head pressure of 40 psi. A 1.0 μL sample was injected in the splitless mode onto a retention gap of approximately 0.5 m methyl deactivated silica (Chrompack, Raritan, NJ).

A Hewlett-Packard 5890 series II gas chromatograph was connected to a HP 5989A mass spectrometer. The carrier gas was helium (Gas Tech, Hillside, IL), and methane (Liquid Carbonic, Chicago) was used as the electron capture, negative ionization (ECNI) reagent gas. The reagent gas pressure was 0.60 Torr, the ion source temperature was 125 °C, and the quadrupole mass analyzer was held at 100 °C. To detect the heptachlor epoxides, ions at *m/z* 354, 386, 388, 390, and 392 were monitored. To detect *cis*- and *trans*-chlordane, ions at *m/z* 374, 376, 406, 408, 410, and 412 were monitored. For the γ-cyclodextrin column, the *m/z* 410 ion was omitted, but all other ions remained the same. These ions included several of the M^- ions of different chlorine isotopomers, as well as $M\text{-}Cl^-$ isotopomer ions.

Peak fitting was necessary to accurately determine the ER for air samples, which often had low *S/N* ratios. The software package used for this purpose was Peak Fit version 4.0 (Jandel Scientific, San Rafael, CA). Abundance and time data were extracted from each data file using HP Chemstation software and copied into Peak Fit, which was set to run and restart as necessary until the r^2 value was no longer changing, suggesting the best fit had been obtained. Peak areas, standard errors, r^2 values, peak centers, and resolution results were then copied to spreadsheets in Microsoft Excel. The fast Fourier transform smoothing function in Peak Fit was chosen to remove high-frequency oscillations in the data. The linear progressive baseline was used in most cases. Occasionally, the presence of another compound required that the linear or constant baseline be used instead. The "vary widths" option was used to allow for two peaks of different width. The peak form chosen to model the chromatographic peaks was the Exponentially Modified Gaussian-Half-Gaussian Modified Gaussian. This peak form models intracolumn effects such as dispersive effects, mass-transfer resistances, axial diffusion, and slow kinetics of adsorption and desorption, all of which tend to broaden peaks (*20*).

Gas-phase air samples were collected for 24 h every 12 days with a high-volume air sampler (Graesby General Metal Works, Smyrna, GA) resulting in a total of approximately 800 m^3 of air. The organochlorine compounds were collected in a stainless steel cartridge containing 30−45 g of Amberlite XAD-2 resin (Sigma, St. Louis, MO), which was Soxhlet extracted with 50% acetone in hexane (EM Science, Gibbstown, NJ) for 24 h. The extract was concentrated, exchanged into hexane, and fractionated on a 3.5 in. long column of silica gel (Aldrich Chemical, Milwaukee, WI) using 25 mL of hexane, 50% hexane in dichloromethane, and dichloromethane. The second fraction was again concentrated before injection.

The samples were collected at the following three sites: *(a)* 48 samples were collected from August 1994 to September 1995 at Sturgeon Point, NY, which is near Lake Erie (latitude 42°41′34″; longitude 79°03′20″); *(b)* six samples were collected August 13, 1994 (with duplicate), January 16, August 9 (with

TABLE 1. Enantiomeric Ratios (ER) and Standard Errors for Standards[a]

	racemic standards		enriched standards	
compd	α-cyclodextrin	γ-cyclodextrin	α-cyclodextrin	γ-cyclodextrin
cis-chlordane	1.017 ± 0.003[b]	1.028 ± 0.003	0.184 ± 0.005	0.170 ± 0.001
	1.010 ± 0.007	1.024 ± 0.006	0.181 ± 0.005	0.167 ± 0.002
	1.001 ± 0.006	1.028 ± 0.007	0.190 ± 0.003	0.169 ± 0.004
	0.996 ± 0.004	1.030 ± 0.007	0.178 ± 0.003	0.175 ± 0.001
	1.010 ± 0.005	1.029 ± 0.008	0.179 ± 0.008	0.166 ± 0.001
	1.007 ± 0.003[c]	**1.028 ± 0.003**	**0.183 ± 0.002**	**0.169 ± 0.001**
trans-chlordane	1.044 ± 0.001	0.967 ± 0.003	16.5 ± 0.1	16.5 ± 0.6
	1.040 ± 0.001	0.984 ± 0.003	17.0 ± 0.1	19.3 ± 0.9
	1.035 ± 0.001	0.981 ± 0.002	17.3 ± 0.1	17.7 ± 0.5
	1.041 ± 0.001	0.968 ± 0.003	16.8 ± 0.1	17.9 ± 0.3
	1.043 ± 0.001	0.986 ± 0.003	17.2 ± 0.1	18.2 ± 0.5
	1.040 ± 0.001	**0.977 ± 0.002**	**17.0 ± 0.1**	**17.9 ± 0.3**
endo-heptachlor epoxide	1.029 ± 0.001		0.118 ± 0.001	
	1.028 ± 0.002		0.116 ± 0.001	
	1.022 ± 0.001	DNS[d]	0.114 ± 0.001	DNS[d]
	1.028 ± 0.001		0.116 ± 0.001	
	1.029 ± 0.001		0.113 ± 0.001	
	1.027 ± 0.001		**0.116 ± 0.001**	
exo-heptachlor epoxide	1.04 ± 0.02	1.033 ± 0.001		47.5 ± 0.4
	1.09 ± 0.02	1.037 ± 0.002		54.0 ± 1.0
	1.04 ± 0.04	1.044 ± 0.001	DNS[d]	48.7 ± 0.3
	1.07 ± 0.03	1.038 ± 0.002		51.0 ± 0.7
	1.07 ± 0.02	1.023 ± 0.002		53.0 ± 2.0
	1.06 ± 0.01	**1.035 ± 0.002**		**51.0 ± 0.8**

[a] ERs are reported as the area of the (+) enantiomer divided by the area of the (−) enantiomer. [b] Standard deviations from the multiple ions that were quantitated. [c] Bold numbers are the average of the five injections given just above. [d] DNS: "did not separate".

duplicate), and August 21, 1995, at Eagle Harbor, MI, which is near Lake Superior (latitude 47°27′47″; longitude 88°08′59″); and *(c)* five samples were collected August 12, 1994, May 17 (with duplicate), July 4, and August 9, 1995, at Sleeping Bear Dunes, MI, which is near Lake Michigan (latitude 44°45′38″; longitude 86°03′30″). These samples were collected as part of the Integrated Atmospheric Deposition Network (IADN) project that studies a variety of compounds being transported to the Great Lakes (*19*). These samples had previously been analyzed for chlordane concentrations, and they had been stored at −20 °C. Samples which had evaporated were made up to 1 mL in hexane before injection.

Results and Discussion

To ensure that reliable ER results could be obtained, a series of standards was analyzed. Figure 2 shows chromatograms of racemic (panels A and C) and enriched (panels B and D) standards separated on both the α- and γ-cyclodextrin (CD) columns. The enriched standards were used to assign peaks to the correct enantiomers. Notice the different separation characteristics on the two columns. The sequence of elution of *cis*- and *trans*-chlordane and *exo*-heptachlor epoxide is reversed, and *endo*-heptachlor epoxide is resolved on α-CD but not on γ-CD. Other researchers have also noted that the elution order of chlordane components reverses on different cyclodextrin phases (*11, 21*). Unfortunately, the interactions occurring between analytes and the cyclodextrins is not fully understood, and these changes in elution sequence cannot be predicted.

The goal of this work was to accurately determine the ratio of the two enantiomers in atmospheric samples by measuring the ratios of the two entantiomerically resolved GC peaks. If the signal is strong because the compound is relatively abundant in the environment, various measures of the peak ratio can provide acceptable results. In fact, some previous studies simply measure the ratio of peak heights (*22, 23*). As the environmental concentration decreases, this approach is no longer adequate, and somewhat more sophisticated peak-fitting techniques are required. This is illustrated in Figure 3 which shows the (−) and (+) peaks monitored at m/z 406 for *trans*-chlordane resolved on α-CD. The three panels represent samples isolated from air near Lake Erie. Figure 3A shows a very good fit resulting from a relatively high chlordane concentration (39 pg/m^3); Figure 3B shows data at a more typical concentration (7.6 pg/m^3); and Figure 3C shows data from a sample with a very low concentration (0.5 pg/m^3). The computer-generated peak fits are given in each case. It is interesting to note that the ERs, based on the ratios of the fitted peak areas, for the first two cases are similar (ER = 0.92 and 0.86), which is a testimony to the power of the peak-fitting approach. We did not consider the data such as shown in Figure 3C reliable, and data with such a poor signal-to-noise ratio were excluded from this study.

TABLE 2. Percentage (and Standard Error) of the (+) Enantiomer in Enriched Standards

compd	experimental[a]	reported[b]
cis-chlordane	15.0 ± 0.2	15.3
trans-chlordane	94.6 ± 0.2	96.3
endo-heptachlor epoxide	10.4 ± 0.1	8.4
exo-heptachlor epoxide	98.1 ± 0.1	97.3

[a] Results for α- and γ-CD were averaged. [b] No error limits were available for these standards.

To verify that the measured ERs were accurate, we carried out two sets of quality control experiments. In the first, we measured the ERs of racemic standards, which should give an ER of 1.00. These data are given in Table 1, left. A word on replication is in order. Each experiment consisted of two injections of the racemic mixture (as illustrated in Figure 2, panels A or C), one injection on α-CD, and one on γ-CD. In each experiment, ions at six different m/z values on the α-CD column, and ions at five different m/z values of the γ-CD column were monitored. Thus, five or six different ERs were determined by peak fitting these different selected ion monitoring records. The average and standard errors of these five (or six) individual ERs are given as an individual entry

TABLE 3. Average ER and Standard Errors for Chlordane Components in Gas-Phase Air Samples Collected near the Great Lakes

	Lake Superior $N = 5$[a]	Lake Michigan $N = 6$	Lake Erie $N = 48$	Lake Ontario	avg[b]
cis-chlordane	1.01 ± 0.03	1.02 ± 0.03	1.12 ± 0.03		1.05 ± 0.02
	1.09 ± 0.03[c]			*1.03 ± 0.03*[c]	
trans-chlordane	0.88 ± 0.02	0.83 ± 0.02	0.93 ± 0.01		0.88 ± 0.02
	0.87 ± 0.03[c]			*0.91 ± 0.03*[c]	
exo-heptachlor epoxide	2.10 ± 0.10	2.00 ± 0.10	2.00 ± 0.03		1.99 ± 0.04
	1.99 ± 0.06[c]			*1.86 ± 0.05*[c]	

[a] Number of samples analyzed. [b] Average including all five data values. The standard error of these five data is given. [c] *Italics* indicate data from ref *15*. The standard error was assumed to be ±3%.

in Table 1, left. For example, the first experiment for *cis*-chlordane on the α-CD column gave an average ER of 1.017 ± 0.003; the first experiment on the γ-CD column gave an average ER of 1.028 ± 0.003. These pairs of injections were repeated five times, and the resulting ERs were averaged. These overall averages and the associated standard errors are shown in bold font in Table 1. These sets of experiments show that the racemic ratios are, on average, within 3% of the true value of 1.00. The *trans*-chlordane values on the α- and γ-CD are oddly on either side of the true value. The enantiomers have been correctly identified, so the origin of this discrepancy is not yet understood. *exo*-Heptachlor epoxide on α-CD is furthest (6%) from the correct racemic value, but this compound is barely separated on this column.

For further quality control, our ER results for enriched standards were compared to the results of Prof. König's laboratory, which had prepared the standards. Although the identification of enantiomers was by optical rotation, his method of quantitation is the same method used here, and no indication of the error of his measurements was provided (*8*). Table 1, right, shows the individual experiments and averages as described above. The comparison of our data (averaged over both columns) with the reported percent enrichment for this mixture is given in Table 2. Our values and the reported values differ by (on average) 1.2%, absolute. We consider this to be excellent agreement, given that both the reported and experimental values have standard errors of about ±3%.

Both chiral columns were used for the analysis of the atmospheric samples. To ensure consistent results, the ERs for *trans*-chlordane were compared for all samples analyzed on both columns. (Although both *cis*- and *trans*-chlordane separated on both columns, it was discovered that endosulfan I coeluted with one of the *cis*-chlordane peaks on γ-CD.) By comparing the mean difference between the paired ER values, it is found there was no statistical difference between the two columns ($t = 1.83$, $\alpha = 0.077$), suggesting that our ER measurements are transferable from one column to the other.

The results of our ER measurements of *cis*- and *trans*-chlordane and *exo*-heptachlor epoxide in air sampled near Lakes Erie, Michigan, and Superior are given in Table 3. *endo*-Heptachlor epoxide was not detected in any samples. All of these ERs are statistically different than unity ($\alpha = 0.05$), except for *cis*-chlordane at Lakes Superior and Michigan. For perspective, we have added ER data from Wiberg et al. (*15*) for air samples taken near Lakes Superior and Ontario to Table 3 (see the ERs given in italics). Unfortunately, no indication of error was given for these data, but we assume that they are accurate to about ±3%, and we have indicated this in Table 3. In general, the agreement between the results of Wiberg et al. and those from this study is excellent.

The ERs for *cis*-chlordane are about the same for air collected near Lakes Superior, Michigan, and Ontario, and these ER values indicate that, for air near these three lakes, *cis*-chlordane is virtually racemic (overall average ER of 1.05 ± 0.02, including values of Wiberg et al.). The ER for air collected near Lake Erie is slightly higher at 1.12 ± 0.03, but it is not clear what might cause this elevation. Perhaps it is because Lake Erie is (on average) the warmest of the Great Lakes, and therefore, this lake has a higher level of biological activity than the others.

The ER for *trans*-chlordane is significantly less than racemic for air collected near all four of the lakes. There are differences among the sites, with the air collected near Lake Erie having the highest ER (0.93 ± 0.01) and the air collected near Lake Michigan having the lowest ER (0.83 ± 0.02). These two ER values, at least, are significantly different from one another for reasons that are not yet clear. Nevertheless, it is clear that the (−) enantiomer of *trans*-chlordane is enriched in these air samples, whereas there was no enantiomeric enrichment (except for Lake Erie) for *cis*-chlordane in these same samples.

One possible explanation of the ER difference between the two chlordane compounds is that the enantiomers are degraded differently in the environment. For example, the first identification of oxychlordane was from pigs fed racemic *cis*- or *trans*-chlordane for 90 days. Measurement of the resulting pig fat showed that racemic *trans*-chlordane produced mostly (+) oxychlordane, but racemic *cis*-chlordane produced racemic oxychlordane (*24*). This result suggests that the metabolic pathways of *trans*- and *cis*-chlordane are different. Other studies with rat liver postmitochondrial supernatant show that 38% of *trans*-chlordane was converted to oxychlordane, while only 14% of *cis*-chlordane was converted to oxychlordane (*25*). Apparently, *trans*-chlordane degrades differently and more rapidly than its *cis*-isomer. More studies are needed to confirm the chiral behavior of these compounds as they degrade.

For *exo*-heptachlor epoxide, the overall average ER is 1.99 ± 0.04 (see Table 3), and the variations between the lakes are probably not significant. Clearly, this compound, which is a metabolite of the pesticide heptachlor, has been very significantly enriched in the (+) enantiomer. This high ER presumably results from the production of heptachlor epoxide through enzymatic processes and not from photolysis alone, which produces small amounts of racemic *exo*-heptachlor epoxide (*5*). Future studies of enantiomeric ratios in the ambient atmosphere should include both oxychlordane as the metabolite of chlordane, and heptachlor as the starting product of *exo*-heptachlor epoxide to gain further insight into these processes.

Others have found that the ER of α-HCH changes seasonally due to volatilization during the warmer months (*26*). However, because our data covered only a limited time period (one year at most), it was not possible to determine if the ERs of chlordane compounds changed as a function of time. In addition, the samples taken in the colder winter months had the lowest chlordane concentrations, and thus, the error among the ERs was a strong function of the atmospheric temperature. For this reason, it was not possible to determine if the ERs of these compounds were a function of atmospheric temperature.

Acknowledgments

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