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Isolation of the Chlordane Compounds U82, MC5, MC7, and MC8 from Technical Chlordane by HPLC Including Structure Elucidation of U82 and Determination of ECD and NICI-MS Response Factors

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A combination of reversed-phase and normal-phase HPLC techniques allowed to isolate amounts in the order of 5−300 μg of the octachloro isomers U82, MC5, MC7, and MC8 from technical chlordane. Their identity was confirmed by capillary gas chromatography mass spectrometry on two stationary phases. ^{1}H NMR and mass spectrometry allowed to elucidate the structure of U82 as 1-*exo*,2-*endo*,3-*exo*,4,5,6,8,8-octachloro-3a,7,7a-tetrahydro-4,7-methanoindane. This chiral isomer is the most abundant octachloro isomer in humans and the only one identified so far having an H atom at the bridge head atom C7. Response factors were determined for negative ion chemical ionization mass spectrometry (NICI-MS) and deviated by a factor of 1.6−2.6 from those for *cis/trans*-chlordane. For electron capture detection (ECD), only MC5 showed a significant difference. Beside the missing Cl atom at C7, the structure of U82 is identical to that of *trans*-nonachlor, which shows a high accumulation and slow metabolism in mammals. This might be the reason for a similar behavior of U82.

Introduction

Technical chlordane is a pesticide consisting of at least 120 compounds (*1*). Though its use has been banned many years ago (e.g., in the United States in 1987), it is still found in the environment. Particularly in the Arctic, chlordane belongs to the most abundant environmental pollutants. Levels in polar bears and Inuits are only exceeded by those of toxaphene and some polychlorinated biphenyls (*2, 3*). Therefore, the quantification of chlordane is still important. Usually, the most abundant congeners in the technical mixture (*cis/trans*-chlordane and *cis/trans*-nonachlor) are quantified together with the main metabolites oxychlordane and *cis/trans*-heptachlorepoxide. All are commercially available as pure reference compounds. Other chlordane congeners were also occasionally quantified (*4*). Since no pure standards are commercially available, for example, averaged response factors of *trans/cis*-chlordane are used. Different acronyms are applied for the less commonly determined chlordanes. The compounds identified by Miyazaki et al. (*5*) are assigned with "MC" as prefix. Those listed as unknowns by Dearth and Hites (*1*) have an "U" in front of the number. The first digit informs about the number of chlorine atoms, and the second one represents the elution order.

The chlordane congeners MC5, MC7, and U82 are also present in percentage quantities in the technical product (6.1% of MC5 and 2.2% of U82 and MC7) (*1, 6*). Their structures are given in Figure 1 together with the numbering of the carbon skeleton. Figure 2 shows that all three compounds can be found in considerable quantities in ambient air, marine biota, and human adipose tissue. In mammals representing the highest trophic level such as seals and humans, U82 and MC5 are the most abundant octachloro congeners (*7*). They show high bioaccumulation factors in the food web. For humans, an accumulation factor of 42 was reported for U82 relative to *cis*-chlordane (*7*). These reports justify analysis of U82 in biota.

Although U82 is an important chlordane congener, its structure was never elucidated. The only partial structure information available so far is the presence of five chlorine atoms at the 6-ring and three at the 5-ring, which was obtained from the retro-Diels−Alder fragment in the mass spectrum (*1, 8*). Furthermore, U82 has been separated into enantiomers, which reveals a chiral structure (*9*).

The original aim of this work was to isolate U82, MC5, and MC7 from technical chlordane and to elucidate the structure of the first one. Since the first experiments showed that MC8 was easy to obtain as well, this compound was later included too. A combination of different high-performance liquid chromatographic separation techniques was considered as the most successful approach. Details about the isolation procedure and structure elucidation are presented. Furthermore, reasons for the high persistence of U82 in mammals are briefly discussed.

Experimental Section

Reference Compounds and Solvents. Technical chlordane and crystalline *cis*-chlordane were obtained from Ehrenstorfer (Germany). The composition of the technical chlordane was similar to that characterized by Dearth and Hites (*1*) and ressembled that from Velsicol Corp., the most applied type of chlordane. *n*-Hexane of HPLC quality (Machler, Switzerland) and acetonitrile of far-UV quality (Romil, U.K.) was employed. Water was obtained from a water purification system combining reversed osmosis, ion exchange, charcoal filtration, and UV treatment (Elga Prima/Maxima, U.K). Water-free Na_2SO_4 for organic trace analysis (Merck, Germany) was used without further treatment. Technical chlordane solutions of about 150 ng/μL were prepared in acetonitrile. A SRM 1588 reference cod liver oil sample (NIST, Gaithersburg, MD) was cleaned up and characterized as described in ref *9* to identify U82, MC5, and MC7 in the isolated fractions.

Isolation by High-Performance Liquid Chromatography. All separations were carried out on a Hewlett-Packard (HP) 1050 high-performance liquid chromatographic (HPLC) system equipped with a diode array detector. Detection was carried out at a wavelength of 240 nm. Depending on the isomer amount to be collected, the overall procedure described below was repeated up to 120 times.

Reversed-Phase HPLC. The first fractionation step of technical chlordane was carried out on a semipreparative

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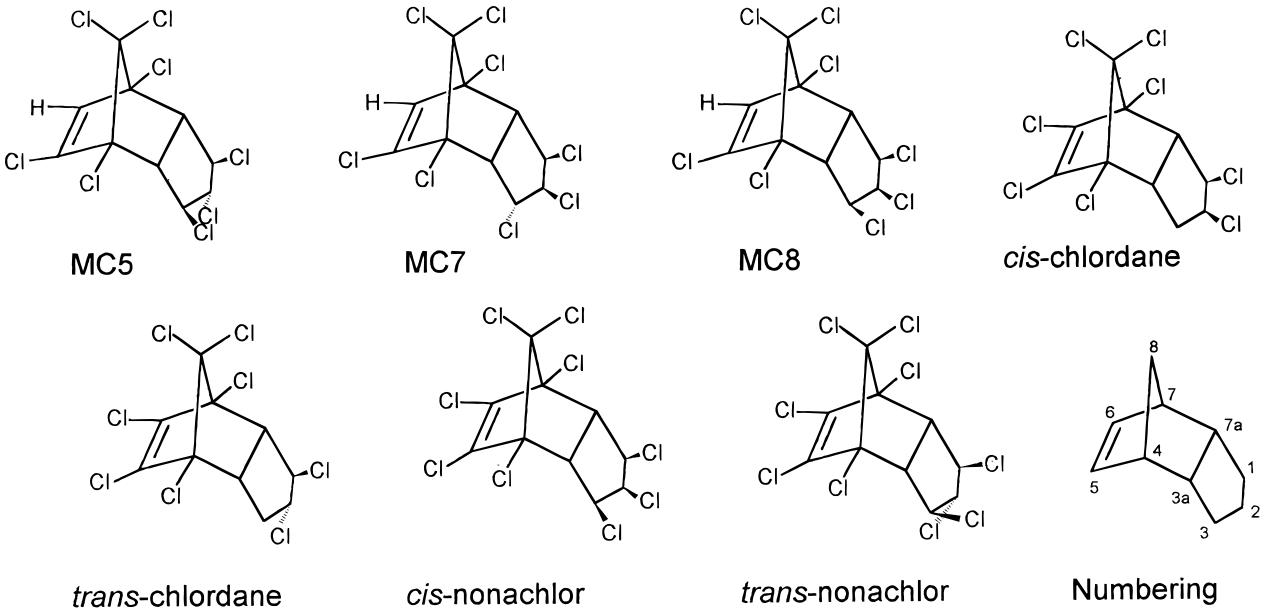


FIGURE 1. Structures of selected chlordane compounds and carbon atom numbering.

C_{18} reversed-phase column of 125 mm length and 10 mm i.d. packed with VP Nucleosil (particle size 7 μm, pore size 10 nm, Macherey-Nagel, Switzerland). A total of 20−50 μL of the technical chlordane solution was injected via a 75-μL sample loop. The following mobile phase gradient was applied at a flow rate of 4 mL/min: acetonitrile/water 60:40 (v/v) for 15 min, then within 20 min to 80:20 followed by a 15-min flushing with pure acetonitrile. Solvents were degassed with helium of 99.9990% purity. One main fraction a1 was collected between 19.5 and 22.3 min containing U82, MC5, MC7, and MC8 (see Figure 3A).

The combined fraction was split into 20 mL volumes. To each aliquot about 3 mL of water and a spoonful of NaCl was added. Then, it was extracted three times with 3 mL of *n*-hexane. After drying over Na_2SO_4, the volume was reduced to 0.5 mL by a TurboVap 500 (Zymark, MS) at fan speed B (5000 min^{-1}) and a water bath temperature of 50 °C. The extracts were combined and the volume reduced further. Then, a further fractionation by normal-phase HPLC was carried out.

Normal-Phase HPLC. Separations were carried out on a silica column of 250 mm length and 4.6 mm i.d. packed with silica (EC Nucleosil, particle size 5 μm, pore size 10 nm, Macherey-Nagel, Switzerland). Between 20 and 50 μL was injected into a 75-μL injection loop. *n*-Hexane was used as the mobile phase at a flow of 1 mL/min. The content of the collected fractions was controlled by GC/MS as outlined below.

To achieve stable retention conditions, *n*-hexane was stirred over water-free Na_2SO_4 for a minimum of 24 h. Then 1000 ppm acetonitrile was added as a modifier. Fractions containing U82 (b1, 8.3−8.7 min), MC5 (b2, 8.7−9.2 min), MC7 (b3, 10.8−11.3 min), and MC8 (b4, 11.3−11.8 min) were collected (see Figure 3B). U82 and MC5 were purified once more using *n*-hexane modified with only 100 ppm acetonitrile. U82 (c1, 12.9−13.6 min) and MC5 (d1, 11.3−11.8 min) were obtained as indicated in Figure 3, panels C and D. The combined fractions were evaporated to 0.5 mL using the TurboVap 500 system.

Gas Chromatography/Mass Spectrometry. GC/MS analysis was carried out on a HP 5890 II gas chromatograph connected to a HP 5989B mass spectrometer. The temperature of the ion source was 200 °C, and that of the quadrupole was 100 °C. An electron energy of 70 or 100 eV was applied for electron impact (EI) and negative ion chemical ionization (NICI), respectively. Methane of 99.995% purity was employed as reagent gas for NICI at an ion source pressure of 0.6 mbar. Full-scan mass spectra were recorded from m/z 30 to m/z 500 with a scan time of 1.25 s. Selected ion monitoring of the octachloro congeners in the SRM 1588 sample was carried out in the NICI mode at m/z 407.8 $(M + 2)^-$ and m/z 409.8 $(M + 4)^-$ using a dwell time of 80 ms per ion.

The high-resolution gas chromatographic (HRGC) separations were performed as follows: Splitless injection of 1 μL; splitless time 2 min; injector temperature 250 °C; transfer line 250 °C; carrier gas, He, at a flow velocity of 35−40 cm/s (180 °C). The following columns were used: 22 m × 0.25 mm i.d. coated with 0.14 μm of polymethylsiloxane (Ultra 1, HP); 30 m × 0.25 mm i.d. capillary coated with 0.1 μm of 90% biscyanopropyl/10% phenylcyanopropylpolysiloxane (RTx-2330, Restek, PA); temperature program for both capillaries, 50 °C for 2 min, then 3 °C/min to 240 °C, isothermal for 2 min. The retention order on the Ultra 1 capillary was used to identify compounds as described by Dearth and Hites (*1*). Enantioselective separations were carried out on a tandem column consisting of a 30 m × 0.25 mm i.d. front column coated with 0.1 μm of RTx-2330 (Restek, PA) coupled to a enantioselective column of 23 m × 0.25 mm i.d. with 0.14 μm of 10% heptakis(2,3,6-*O*-*tert*-butyldimethylsilyl)-β-cyclodextrin in PS086 (15% phenyl/85% dimethylpolysiloxane, OH terminated) (*9*). Compound identification was carried out as described in ref *9*. The temperature program was 90 °C for 2 min, then 15 °C/min to 180 °C, isothermal for 44 min, 2 °C/min to 230 °C, and isothermal for 2 min. The separations shown in Figure 2, panels C and D, were achieved with the two last tandem system in Table 3 of ref *10*.

^{1}H NMR Spectra. A Bruker ARX spectrometer was used operating at 500.13 MHz. Chemical shifts were determined relative to the solvent signal ($CDCl_3$) at 7.26 ppm. One-dimensional spectra were recorded at 4, 22, and 45 °C to optimize peak separation. Homo decoupling experiments were performed to assign the coupling partners in the spin system. Neighborhood correlations were partly established by two-dimensional (2D) nuclear overhauser enhancement spectroscopy (NOESY). The 2D spectrum was recorded with 640 increments and 4096 data points on each increment. The total accumulation time was 38 h, and the mixing time was 200 ms. The spin system was computer simulated using the Pearch program package (Department of Chemistry, University of Kupio, Finland).

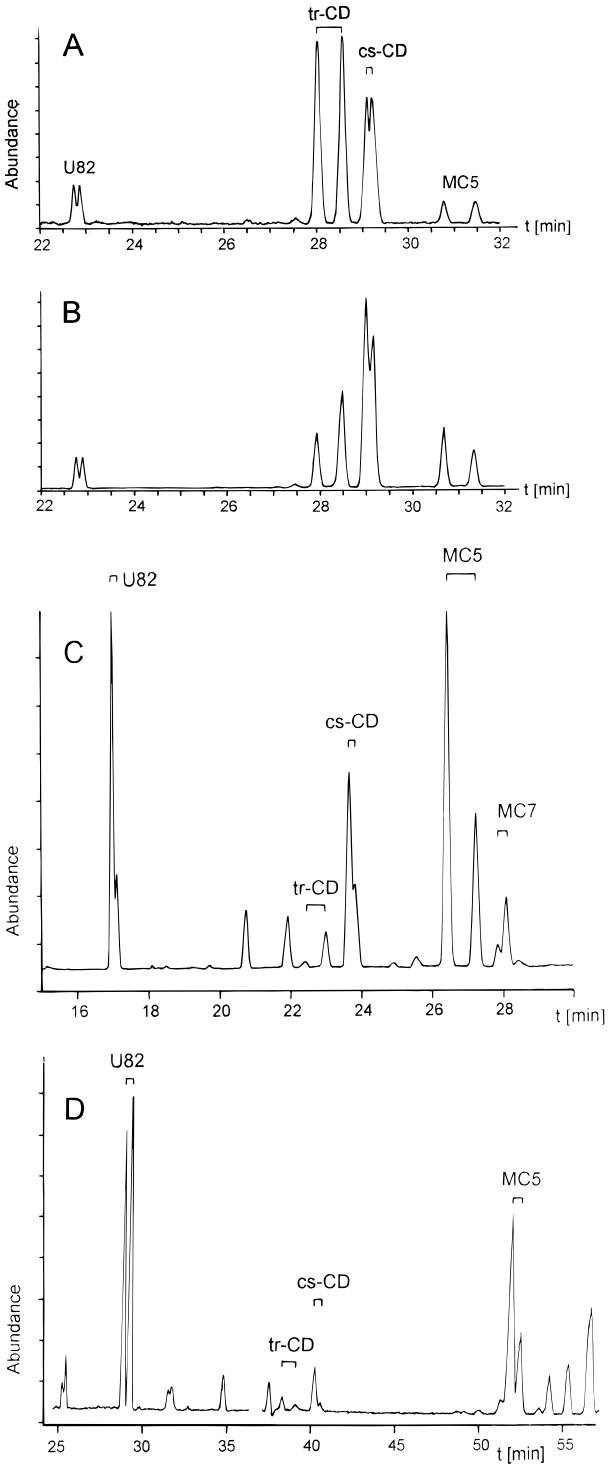


FIGURE 2. Presence of octachloro chlordane compounds in (A) ambient air, (B) herring, (C) seal blubber, and (D) human adipose tissue. The NICI mass chromatograms of *m/z* 409.8 of the separation on the enantioselective column are shown. Chromatograms C and D were obtained with the two last tandem column systems and temperature programs described in Table 3 of ref *10*.

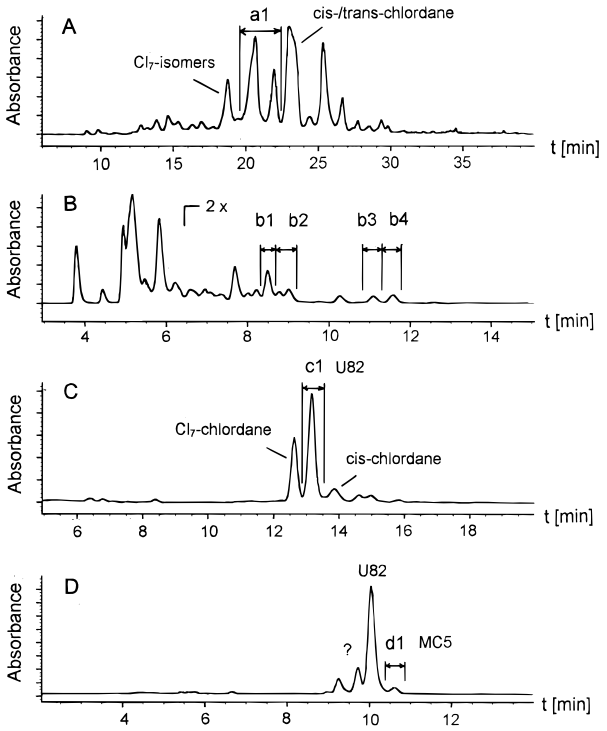


FIGURE 3. HPLC chromatogram of the isolation of U82, MC5, MC7, and MC8. (A) Reversed-phase HPLC with acetonitrile/water 60:40. Fraction a1 contained all target isomers. (B) Normal-phase HPLC with *n*-hexane/1000 ppm acetonitrile with the four collected fractions b1 (U82), b2 (MC5), b3 (MC7), and b4 (MC8). (C) Normal-phase HPLC with *n*-hexane/100 ppm acetonitrile to isolate fraction c1 with U82. (D) Normal-phase HPLC *n*-hexane/100 ppm acetonitrile to obtain fraction d1 with MC5.

Flame Ionization and Electron Capture Detection Response Factors. A HP 5890 gas chromatograph equipped with a flame ionization detector (FID) at 250 °C was used to quantify the isolated amounts of U82, MC5, MC7, and MC8 against *cis*-chlordane, which has the same carbon skeleton structure and elemental composition and, therefore, the same response factor. A hydrogen flow of 35 mL/min and an air flow of 350 mL/min were employed. The electron capture response factors were measured on a HP 6890 gas chromatograph equipped with a ^{63}Ni electron capture detector (ECD) kept at 250 °C. Nitrogen at a flow rate of 100 mL/min was used as moderation gas. The ratio anode flow to moderation gas flow was 1:10.

Results and Discussion

Purity and Characterization of Isolated Chlordane Isomers. The reversed-phase HPLC cleanup allowed the removal about 90% of the chlordanes of no interest. A major problem was to eliminate the remaining quantities of *trans*-chlordane and of some other congeners. This could only be achieved by normal-phase chromatography. Stable retention conditions could be obtained by adding acetonitrile as a polar modifier to the dried mobile phase (*11*). Two normal-phase HPLC steps were necessary to remove remaining chlordane congeners; the second one with the lowest possible amount of acetonitrile. The compound purity was determined by HRGC on two different phases (see Experimental Section) using NICI-MS and was as follows: U82, 96% (4% of *trans*-chlordane and traces of a heptachloro congener); MC5, 90% (impurities due to two compounds with similar retention time as U82 but different mass spectra as well as a further trace constituent not identified); MC7, >99%; MC8, >99%. About 300 μg of U82, 5 μg of MC5, 50 μg of MC7, and 50 μg of MC8 were isolated. Figure 4 shows the NICI mass chromatograms of the isolated compounds obtained on the enantioselective tandem column and compares them with the octachloro congener pattern in the SRM1588 cod liver oil extract.

Structure Elucidation of U82. The EI mass spectrum is shown in Figure 5. A molecular ion of low abundance was present at *m/z* 406. Fragments were formed by consecutive

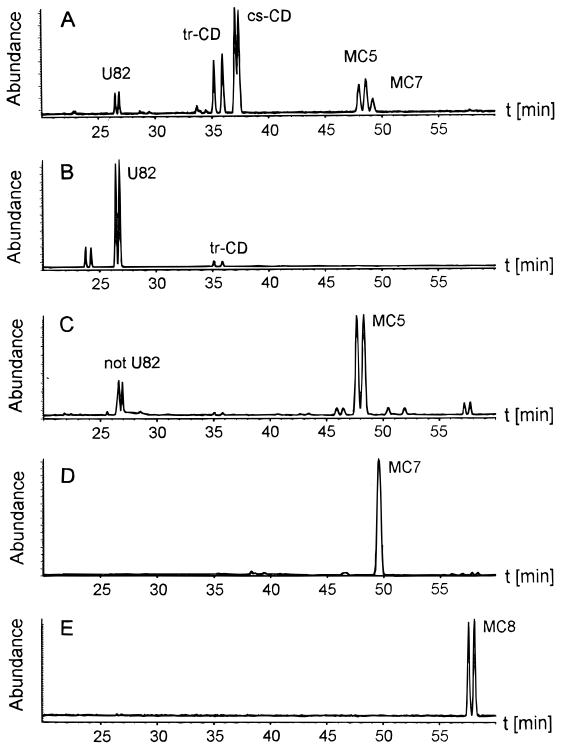


FIGURE 4. NICI mass chromatograms of *m/z* 407.8 (full-scan mode except panel A) of the isolated octachloro isomers obtained with the enantioselective tandem column. (A) Cod liver oil extract (selected ion monitoring). (B) U82 fraction c1. (C) Fraction d1 containing MC5. (D) MC7 fraction b3. (E) MC8 fraction b4. tr-CD, cs-CD: *cis/trans*-chlordane.

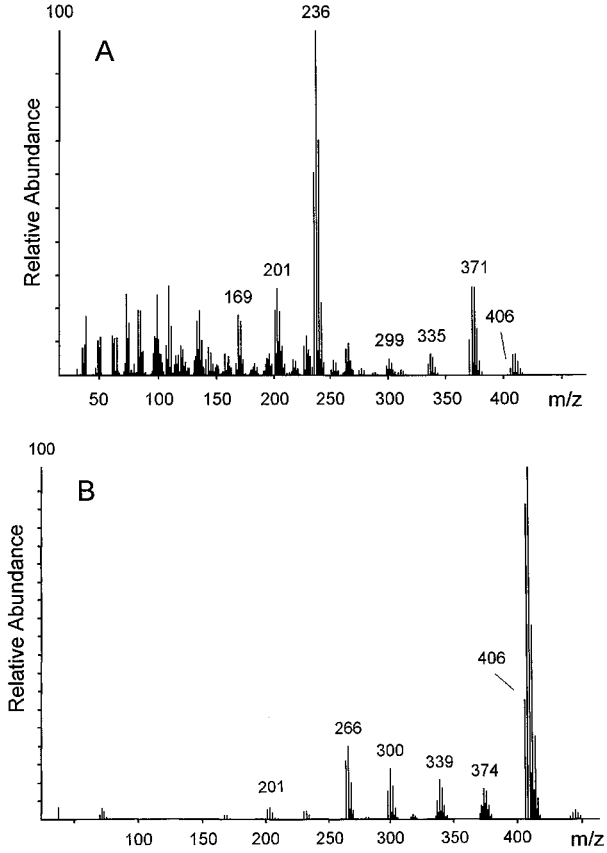


FIGURE 5. EI (A) and NICI (B) mass spectra of U82.

losses of Cl or HCl leading to *m/z* 371, 335, and 299, for example. The most abundant ion at *m/z* 236 $(C_5Cl_5H)^+$ is formed by a retro-Diels–Alder cleavage. It determined the distribution of the chlorine atoms in the ring systems. The 6-ring contains five Cl atoms leading to a "5+3" structure (see also refs *1* and *8*). The NICI mass spectrum (Figure 5) was quite similar to that reported in ref *7*. In both spectra the molecular ion (*m/z* 406, base peak) and fragments due to loss of Cl or HCl or exchange of Cl by H were observed such as *m/z* 372 $(M - Cl + H)^-$, *m/z* 337 $(M - 2Cl + H)^-$, *m/z* 298 $(M - 3Cl - 3H)^-$, *m/z* 264 $(M - 4Cl - 2H)^-$, and *m/z* 201 $(C_5Cl_4H)^-$. However, as usual for registration on two different instruments, the abundances deviated.

Figure 6 shows the ^{1}H NMR spectrum of U82. As already found by MS, six protons are present. The observed chemical shifts and coupling constants are summarized in Table 1 and compared with those published for MC5 (a 5+3 structure as well) and *cis*-chlordane (a "6+2" structure) (*5, 12*). All chemical shifts were <4.2 ppm, proving the absence of olefinic protons that have shifts around 6 ppm in chlordanes (*5, 12*). Furthermore, U82 has no geminal protons since no signals appear at chemical shifts <3.4 ppm, and all coupling constants in the ^{1}H NMR spectrum were below 11 Hz. In chlordane structures, geminal protons are typically in the range of 1.5–2.6 ppm with coupling constants of >14 Hz (*5, 12*).

Decoupling H2 at 4.06 ppm revealed H1 and H3 as spin coupling partners. The spin coupling partnership was further confirmed between H2 and H3 by decoupling H3 at 3.59 ppm. Partnership could also be established between H7 and H7a by decoupling H7 at 3.63 ppm. Decoupling H3a at 3.47 ppm affected the spin system of H3.

The information extractable from the 2D NOESY spectrum was limited. Signal overlap made the interpretation of the spectrum rather complex. Nevertheless, neighborhood correlations could be observed between H2 and H1, H2 and H3, H3 and H3a, as well as H3a and H7a.

The isolated double doublet (dd) at 4.06 ppm is characteristic for a proton at C2 in exo position (see also the chemical shifts for MC5 in Table 1). A corresponding endo proton would give a signal more downfield at 4.5 ppm (see *cis*-chlordane in Table 1). Coupling constants of $J = 8.3-10.3$ Hz indicate that the protons on C1, C2, and C3 are positioned endo–exo or exo–endo to each other. Vicinal coupling constants for protons in endo–endo conformation are significantly smaller (e.g., $J = 4.1$ Hz for H2 in *cis*-chlordane, see Table 1 and refs *5* and *12*).

Vicinal coupling constants of $J_{12} = 10.3$ Hz and $J_{23} = 10.2$ Hz were measured between H1 and H2-exo and between H3 and H2-exo, respectively. This implies that H1 and H3 are positioned endo. The chemical shifts of H1-endo, H2-exo, and H3-endo support this conclusion further (see MC5). H1-endo and H3-endo couple further with H7a and H3a, respectively. The coupling constants between these protons was determined as ~8 Hz, revealing an exo configuration of H3a and H7a. The simulation of the ^{1}H NMR spectrum provided a vicinal coupling constant of 9.8 Hz between H3a-exo and H7a-exo, confirming that these protons are orientated in exo–exo configuration. H7a-exo showed an additional coupling constant of 4.0 Hz to the proton H7 at 3.63 ppm. H3a-exo had no additional coupling constants, which excludes any protons on carbons C4 and C8.

The overall NMR information allowed us to define the structure of U82 as 1-*exo*,2-*endo*,3-*exo*,4,5,6,8,8-octachloro-3a,7,7a-tetrahydro-4,7-methanoindane. This structure is in agreement with the mass spectrometric information of a 5+3 chlorine atom distribution between the 6- and 5-rings. Furthermore, the elucidated structure is chiral, which is in accordance with the enantioselective separations shown in Figures 2 and 4. In opposite to most chlordanes, U82 is not chlorinated at the bridge head atom C7 (see Chart 1). Finally,

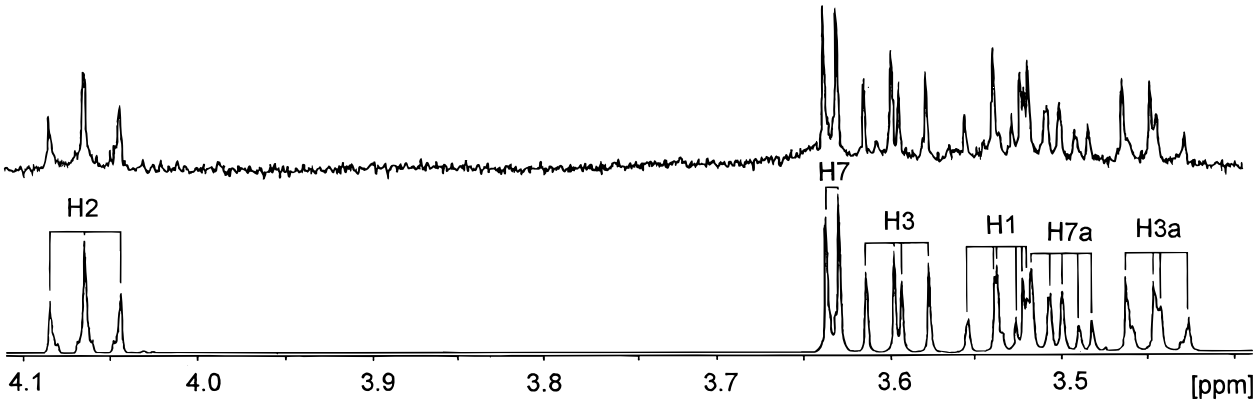


FIGURE 6. Recorded (A) and simulated (B) ^{1}H NMR spectrum of U82.

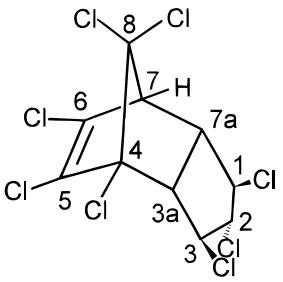
TABLE 1. ^{1}H NMR Chemical Shifts and Coupling Constants for U82 (Measured and Simulated) and Other Selected Chlordanes

	Chemical Shifts (ppm)							
compound	H1 (endo)	H2	H3 (endo)	H3 (exo)	H3a	H7a	H5	H7
U82 measured[a]	3.56−3.51 (m)	4.06 (t)	3.59 (dd)		3.44 (dd)	3.50 (m)		3.63 (d)
U82 simulated	3.53 (m)	4.06 (dd)	3.60 (dd)		3.45 (m)	3.51 (m)		3.63 (d)
MC5[b]	3.61−3.70 (m)	4.09 (t)	3.61−3.70 (m)		3.50−3.53 (m)	3.50−3.53 (m)	6.36 (s)	
cis-chlordane[b]	3.97 (dd)	4.44 (m)	1.82 (m)	2.43 (m)	3.73 (m)	3.55 (dd)		

	Coupling Constants (Hz)								
compound	J_{12}	J_{17a}	J_{23} endo	J_{23} exo	$J_{3enod3a}$	J_{3exo3a}	$J_{3exo3exo}$	J_{77a}	J_{7a3a}
U82 measured[a]	m	m	10.2		8.3			3.8	8.4−9.8
U82 simulated	10.3	8.8	10.2		8.4			4.0	9.8
MC5[b]	m	m	m		m				m
cis-chlordane[b]	4.1	8.1	4.5	2.2	8.9	8.2	14.7		m

[a] In $CDCl_3$ with TMS as the reference; doublet centers are reported; d, doublet; dd, doublet of doublets; t, triplet; m, multiplet. [b] Literature values obtained from refs *5* and *12*.

CHART 1



to the best of our knowledge, U82 does not belong to the chlordane congeners that have been obtained by photochemical synthesis such as MC1, MC2, MC3, and MC5 (*13*).

Determination of ECD and NICI-MS Response Factors. Since no pure standards were available for U82, MC5, and MC7, these compounds have been quantified occasionally using an average ECD or NICI-MS response factor of *cis*/*trans*-chlordane. The response factors for both NICI mass spectrometry and electron capture detection can vary considerably between isomers (a factor of 2−5, see for example ref *14*). On the opposite side, the response of *cis*/*trans*-chlordane is nearly identical when employing mass spectrometry in the EI mode. However, EI-MS is hardly used for chlordane quantification. A substantial fragmentation is observed leading to molecular and fragment ions of low abundance and/or with similar masses as toxaphenes that are present in the same cleanup fraction.

The response of U82, MC5, MC7, and MC8 was determined for ECD and NICI-MS. Quantification of the isolated amounts was carried out by FID, which has identical response factors for isomers (*15, 16*). Within the margin of error, the ECD response of U82, MC7, and MC8 was identical to that of *cis*-chlordane (see Table 2). However, a considerably lower response was found for MC5. This was surprising since the only structural difference between MC5 and MC7 is the exchange of endo/exo positions at C2 and C3. No reasonable explanation can be given for this deviation. The NICI response of all isolated isomers was much higher than for *cis*-chlordane. Therefore, a quantification of U82, MC5, MC7, and MC8 on the basis of the response of *cis*- and/or *trans*-chlordane will lead to an overestimation by a factor of 2−2.5.

TABLE 2. ECD and NICI-MS Response Factors of the Isolated Octachloro Congeners Relative to *cis*-Chlordane Using the Conditions Listed under Experimental Section

compound	ECD	NICI-MS
U82	1.06	0.95
trans-chlordane	0.98	1.61
MC5	0.66	2.08
cis-chlordane	1	1
MC7	1.07	2.61
MC8	0.98	1.99

The NICI response factors relative to *cis*-chlordane were very stable. Changes of instrumental conditions or the separation phase had a influence maximum of 5%, which corresponds to the measuring error. Therefore, the factors given in Table 2 can be used to correct results obtained by quantification with *cis*/*trans*-chlordane on the same instrument, for example.

Metabolic Stability of U82. U82 has an endo chlorine atom at C2 and exo chlorines at C1 and C3. *trans*-Nonachlor and MC5 have a similar configuration. Both accumulate more

in human tissue than *cis*-nonachlor or MC7 (*7*, *17*). This might be one reason for the high accumulation of U82 in human tissue relative to technical chlordane (see ref *7*), which is only exceeded by U81 and U83. *cis*-Chlordane and MC7 have the chlorine atom at C2 in exo position and do not show a significant accumulation to technical chlordane (*7*). In addition, *trans*-chlordane with an endo hydrogen atom at C2 and an exo chlorine atom at C1 also does not accumulate strongly in biota. This demonstrates the importance of a C1-endo, C2-exo, C3-endo chlorine atom constellation for bioaccumulation.

The dechlorination of *trans*-nonachlor into *trans*-chlordane was determined to be the rate-limiting step of the metabolism in humans and rats (*18*). U82 has exactly the same Cl atom configuration as *trans*-nonachlor (only the Cl at C7 is missing). This might be an explanation for the slow metabolism of U82 in rats (half-life of 16 days), which is comparable to that of *trans*-nonachlor (15 days) (*19*). Only U81, U82 and MC6 (= nonachlor III) have longer half-lives (>30 days).

A metabolic formation of U82 from *trans*-nonachlor is not very likely. Biogenic dechlorination of chlordane compounds usually occurs in position C3 (*18*) while photo dechlorination takes place in position C5 (*5*, *13*). Dechlorination at C7 has never been reported.

Conclusions

The presented isolation is a feasible way to isolate larger amounts of the octachloro congeners U82, MC5, MC7, and MC8 from technical chlordane. However, a real preparative system has to be used to obtain larger quantities. The necessity of the availability of U82, MC5, and MC7 as pure reference compounds is supported by their abundant levels in mammals and the considerable differences in their response factors when applying NICI mass spectrometry or electron capture detection (only valid for MC5). The structure elucidation of U82 confirmed a structure similar to *trans*-nonachlor except that the Cl at C7 is missing. This might explain its high persistency. So far, U82 is the only identified octachloro compound having an H instead of a Cl at the bridge head atom C7.

Acknowledgments

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