

Synthesis and Crystallography of 1,2,3,4,6,7-Hexachloronaphthalene and 1,2,3,5,6,7-Hexachloronaphthalene

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Jakobsson, E., Eriksson, L. and Bergman, Å., 1992. Synthesis and Crystallography of 1,2,3,4,6,7-Hexachloronaphthalene and 1,2,3,5,6,7-Hexachloronaphthalene. – *Acta Chem. Scand.* 46: 527–532.

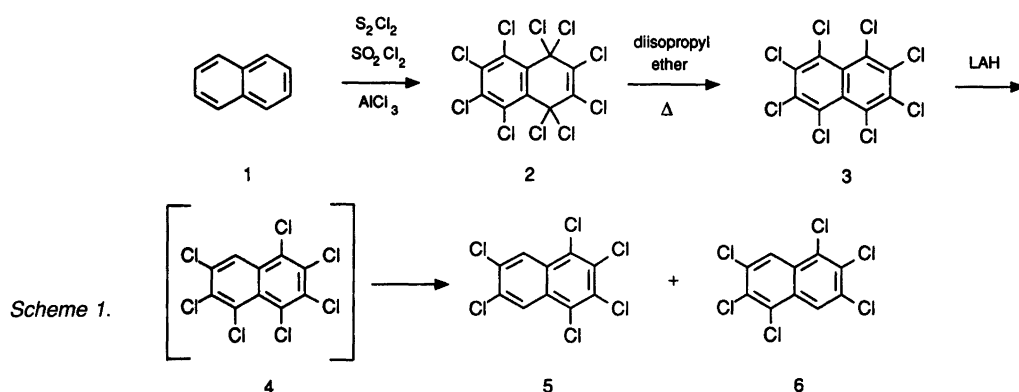
1,2,3,4,6,7-Hexachloronaphthalene (**5**) and 1,2,3,5,6,7-hexachloronaphthalene (**6**) have been synthesized via reductive dehalogenation of octachloronaphthalene by lithium aluminium hydride. The reduction proceeds via the substitution of one of the chlorine atoms in an α -position and the formation of 1,2,3,4,5,6,7-heptachloronaphthalene (**4**). The two sterically more crowded α -positions (positions four and five) in the heptachloronaphthalene were found to be preferentially reduced as the reduction proceeds. Only trace amounts of other isomers were formed. Crystal and molecular structure of the two title isomers were determined by single crystal X-ray diffraction methods. The compounds crystallize in the space group $P2_1/c$ with the following cell dimensions: **5**, $a = 14.71(2)$ Å, $b = 3.828(4)$ Å, $c = 20.45(3)$ Å and $\beta = 87.8(1)^\circ$; **6**, $a = 9.381(4)$ Å, $b = 3.807(2)$ Å, $c = 16.168(8)$ Å and $\beta = 102.40(4)^\circ$. The structures were determined by direct methods and the obtained models containing a total of 95 parameters for **5** and 48 parameters for **6** were refined by full-matrix least-squares calculations that gave a final R value of: **5**, 0.0619 for 486 unique reflections with $I/\sigma(I) \geq 3$ and **6**, 0.0592 for 382 unique reflections with $I/\sigma(I) \geq 3$.

Polychlorinated naphthalenes (PCN), like polychlorinated biphenyls (PCB), have been industrially produced because of their high chemical and thermal stability. PCB and PCN have been used chiefly in the electrical industry, e.g. as separators in storage batteries, as capacitor impregnants, and as seals for condensers and coils. PCN have also been used as additives in oils to clean sludge and petroleum deposits from engines and owing to their flame resistance, fungicidal and insecticidal properties, they have been used to impregnate wood, paper and textiles.¹

PCN are waxy solids that are commercially produced via the chlorination of naphthalene with chlorine gas in the presence of catalytic amounts of iron(III) or antimony(V)

chloride.¹ The production has declined, at least in part, owing to the fact that workers exposed to PCN have suffered serious occupational health problems.

PCN products are complex mixtures with various degrees of chlorination. Toxicological studies of the products indicate that the toxicity increases with the degree of chlorination. In particular the penta- and hexa-chlorinated naphthalenes (pentaCNS/hexaCNS) have been associated with skin problems (Chloracne) and liver damage.^{1,2} Asp-lund *et al.* have reported one of the components in the commercial PCN mixture Halowax 1014 to be strongly retained in rat adipose tissue and liver.³ Considering the GLC retention time, the mass spectrum and the behavior



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on a charcoal column, these authors suggested the structure of the compound to be 1,2,3,5,6,7-hexachloronaphthalene (**6**), Scheme 1.

Over the last few years PCN have been increasingly recognized as a potentially serious environmental problem. In the Swedish environment some tetraCNs and a few pentaCNs and hexaCNs have been reported from analyses of fish samples and mammals.⁴ The predominating HexaCN in the biological samples has a GLC retention time corresponding to the hexaCN in Halowax 1014 with the tentative structure 1,2,3,5,6,7-hexaCN (**6**).⁵

The aim of the present study was to synthesize 1,2,3,5,6,7-hexaCN (**6**) in order to verify its occurrence in the environment, to make it available for toxicological studies and to determine the physical properties of the compound. During the progress of this work we found that there are two possible isomeric hexaCNs that might correspond to the hexaCN found in the Swedish environment. Syntheses of the two isomers and their crystallographic characteristics are presented.

The preparation of several individual CNs dates back to the nineteenth century.¹ In most syntheses, the appropriate chloronaphthalenesulfonyl chloride was heated with phosphorus(V) pentachloride at ca. 200 °C.⁶ The required sulfonyl chloride was often obtained by treating a suitable less-chlorinated PCN with chlorosulfonic acid. However, the exact structures of several of the products have not been fully elucidated.^{1,6} Hitherto only three of the ten possible hexaCNs have been synthesized and described namely, 1,2,4,5,6,8-hexaCN,⁷ 1,2,3,6,7,8-hexaCN⁸ and 1,2,3,4,5,6-hexaCN.⁹

Experimental

Chemicals. Naphthalene was purchased from Janssen Chimica, sulfur monochloride from Aldrich and aluminium chloride, lithium aluminium hydride (LAH) and silica gel (Kieselgel 60, <0.063 mm) were from Merck. Sulfuryl chloride, tetrahydrofuran, diisopropyl ether and hexane (all from Merck) were distilled. Other solvents used were of analytical grade. Thin layer chromatography (TLC) was performed on silica gel plates (Merck, DC Fertigplatten, Kieselgel 60 F254) with hexane as the mobile phase.

Instruments. Gas liquid chromatography (GLC) was performed using a Shimadzu GC-9AM gas liquid chromatograph equipped with a DB-5 fused silica capillary column, 30 m × 0.25 mm, 0.025 mm film thickness (J&W Scientific) and a flame ionisation detector (FID). Temperature program: 80 °C (1 min); 30 °C min⁻¹; 200 °C; 6 °C min⁻¹; 250 °C. The temperatures of the injector and the detector were 210 and 310 °C, respectively.

Gas liquid chromatography–mass spectrometry (GLC–MS) analysis was performed on a Finnigan 4021 instrument upgraded with a 4500 ion source and equipped with an Inco data system. Helium was used as the carrier gas. Electron impact (EI) was used as the ionization method at

an ion source temperature of 140 °C and an electron energy of 70 eV. A GLC column similar to that described above was used and the parameters were the same except for the injector temperature which was held at 240 °C.

High-performance liquid chromatography (HPLC) was performed on a Shimadzu 4-A instrument equipped with a Rhenodyne 7120 injector and a UV spectrophotometric detector (Shimadzu SPD-2AS). The conditions for these separations are described in detail below. UV spectra were recorded in hexane using a Hitachi U-3200 Spectrophotometer.

¹H and ¹³C NMR spectra were recorded for CDCl₃ solutions with a JEOL EX270 instrument at 270 MHz and room temperature.

The crystallographic investigations were performed on a Siemens STOE/AED2 diffractometer equipped with a graphite monochromator to reflect CuK_α radiation.

WARNING! 1,2,3,4,6,7-Hexachloronaphthalene (**5**) and 1,2,3,5,6,7-hexachloronaphthalene (**6**) may have toxicological properties similar to 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD).¹⁰

Decachloro-1,4-dihydronaphthalene (2). A solution of naphthalene (1.0 g, 7.8 mmol, **1**) and sulfur monochloride (0.3 ml, 3.7 mmol) in sulfuryl chloride (25 ml) was slowly added to a boiling solution of sublimed aluminium chloride (250 mg, 1.9 mmol) in sulfuryl chloride (75 ml) according to Ballester *et al.*¹¹ The volume of sulfuryl chloride slowly decreased during the reaction and it was necessary to add further sulfuryl chloride in order to keep the volume constant. After 5 h the sulfuryl chloride was distilled off, the residue was diluted with water (50 ml) and solid sodium hydrogen carbonate was added gradually until no more gas was evolved. The solution was then acidified with concentrated hydrochloric acid and finally extracted with chloroform. The product was purified on an open silica gel column using hexane as the mobile phase and then recrystallized from benzene. Yield 2.9 g (6.1 mmol; 79 %) of white prisms. M.p. 205–210 °C (decomp.). ¹³C NMR (270 MHz, CDCl₃): δ 139.1, 134.2, 132.6, 131.0, 79.7 Hz. Found: C 25.3; Cl 74.7. Calc. for C₁₀Cl₁₀: C 25.3; Cl 74.7. UV [hexane (log ε)]: 231 (5.9), 211 (5.9) nm.

Octachloronaphthalene (3). Decachloro-1,4-dihydronaphthalene (118 mg, 0.25 mmol, **2**) was dissolved in freshly distilled diisopropyl ether (15 ml) and refluxed for 10–36 h during which time **2** was converted into **3**.¹² The reaction was followed by HPLC equipped with a C₁₈ reversed-phase column (Vydac 201 TP, 0.010 mm particles, 10 × 250 mm). Methanol–water (85:15) was used as the mobile phase at a flow rate of 2.5 ml min⁻¹. The retention times of **2** and **3** were 10.5 and 19.9 min, respectively. The time needed for the complete conversion of **2** into **3** varied between 10 and 36 h. The solvent was evaporated off and the product was recrystallized from benzene. The yield of **3** was 80.6 mg (0.21 mmol; 81 %) of light yellow needles. M.p. 198.5–200 °C. MS: *m/z* (rel ab., %): 400 (37, M⁺), 402 (95), 404

(100), 406 (68), 419 (7), 330 [35 ($M-2Cl$)⁺], 260 [32, ($M-4Cl$)⁺], 225 [13, ($M-5Cl$)⁺], 190 [39, ($M-6Cl$)⁺], 155 [17, ($M-7Cl$)⁺]. ¹³C NMR (270 MHz, CDCl₃): δ 135.4, 129.8, 129.1 Hz. UV [hexane (log ε)]: 276 (5.1) nm.

1,2,3,4,6,7-Hexachloronaphthalene (5) and 1,2,3,5,6,7-hexachloronaphthalene (6). Octachloronaphthalene (82 mg, 0.20 mmol, **3**) and lithium aluminium hydride (LAH) (132 mg, 3.5 mmol) were stirred in tetrahydrofuran (75 ml) at room temperature under an argon atmosphere. Water (20 ml) was carefully added after 1 h. The reaction was acidified with hydrochloric acid (3 ml, 5 M) and stirred for an additional hour. After extraction with chloroform (3 × 25 ml), the organic phase was dried with sodium sulfate and after evaporation of the solvent, 90 mg of light brown crystals remained. According to GLC (FID) data, the product consisted of a total amount of 59% of the hexachloronaphthalenes **5** and **6**, 6% of other HxCNs and 35% of lower-chlorinated CNs.

The product was purified from polar by-products on a silica gel column (1 × 10 cm) using hexane as the mobile phase, Fig. 1. In order to isolate the major hexaCNs (**5** and **6**) from the less-chlorinated CNs and trace amounts of other hexaCNs, the mixture was fractionated by HPLC using a charcoal column (Amoco Carbon PX-21 A, 460 × 2.3 mm). The mixture was dissolved in hexane (25 ml) and transferred to the column by 0.5 ml injections. After elution with hexane (100 ml) the solvent was changed to toluene over a period of 60 min. Fractions (5.25 ml) were collected at a flow rate of 0.75 ml min⁻¹. Each fraction was analyzed by GLC (FID) and fractions consisting of more than 85% of the hexaCNs **5** and **6** were pooled. The solvent was evaporated and the mixture was recrystallized from methanol.

Finally, **5** and **6** were separated using HPLC and a 2-(1-pyrenyl)ethyl(dimethyl)silylated (PYE) silica column¹³

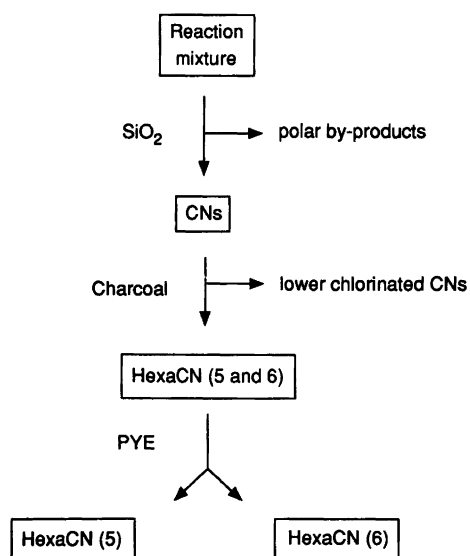


Fig. 1. Isolation procedure.

(10 × 250 mm, particle size 5 μm, Nacalai Tesque, Japan) with hexane as the mobile phase (flow rate 3.0 ml min⁻¹). The mixture of the two hexaCNs was dissolved in hexane and injected in 300 μl portions (250 μg). Fractions were collected manually.

1,2,3,4,6,7-HexaCN (5): MS: m/z (rel ab., %): 332 (51, M^+), 334 (100), 336 (99), 338 (38), 340 (13), 297 [5, ($M-Cl$)⁺], 262 [26, ($M-2Cl$)⁺], 227 [8, ($M-3Cl$)⁺], 192 [14, ($M-4Cl$)⁺]. ¹³C NMR (270 MHz, CDCl₃): δ 133.1, 130.4, 128.2, 128.0, 125.7 Hz. ¹H NMR (270 MHz, CDCl₃): δ 8.40 Hz. UV [hexane (log ε)]: 249 (5.1) nm.

1,2,3,5,6,7-HexaCN (6): MS: m/z (rel ab., %): 332 (52, M^+), 334 (100), 336 (92), 338 (38), 340 (8), 297 [6, ($M-Cl$)⁺], 262 [31, ($M-2Cl$)⁺], 227 [10, ($M-3Cl$)⁺], 192 [22, ($M-4Cl$)⁺]. ¹³C NMR (270 MHz, CDCl₃): δ 133.4, 130.4, 129.1, 127.7, 124.6 Hz. ¹H NMR (270 MHz, CDCl₃): δ 8.34 Hz. UV [hexane (log ε)]: 249 (5.1) nm.

Crystallography. Crystals of the title compounds **5** and **6** were formed by recrystallization from methanol. Systematic absences $h0l:l = 2n+1$ and $0k0:k = 2n+1$ indicated the space group $P2_1/c$ (No. 14) in both cases. Lattice parameters were refined by least-squares calculations based on setting angles of 50 reflections in **5** and 22 selected reflections in **6**. The intensities were collected at room temperature on a Siemens STOE/AED2 diffractometer equipped with a graphite monochromator to reflect Cu K_{α} radiation. The data were corrected for Lorentz and polarisation effects. The crystals of **5** were extremely thin needles and an attempt to correct for absorption effects, using numerical integration ($\mu = 109.8 \text{ cm}^{-1}$) failed to give any improvement and was not used. In the second case (**6**) the crystals were slightly larger and the absorption correction was successfully applied ($\mu = 131.8 \text{ cm}^{-1}$) in this second case. Crystal data for both compounds are found in Table 1.

Results and discussion

1,2,3,4,6,7-HexaCN (5) and 1,2,3,5,6,7-hexaCN (6) were synthesized via reductive dehalogenation of octaCN (**3**) by LAH, cf. Scheme 1.

We have reported the reduction of decachloro-1,4-dihydronaphthalene (**2**) by LAH to give one major hexaCN with the tentative structure 1,2,3,5,6,7-hexaCN (**6**).¹⁴ However, the reduction of **3**, instead of **2**, was found to give a much better yield. Compound **3** can easily be obtained by reflux of **2** in diisopropyl ether.¹² The time necessary for the conversion of **3** into **2** varied between 10 and 36 h. The progress of the reaction was followed by HPLC. The retention times of **2** was about half that of **3** which is consistent with the relative retention time reported by Ivanov *et al.*¹⁵ It was necessary, however, for the wavelength of the UV detector to be changed between the two eluting compounds as **2** and **3** have different absorption maxima. It is not possible to follow the reaction by GLC since **2** decomposes to **3** at temperatures higher than its melting point.¹⁶

Table 1. Crystal data and structural parameters.

	5 (1,2,3,4,6,7-hexachloronaphthalene)	6 (1,2,3,5,6,7-hexachloronaphthalene)
Space group	<i>P</i> 2 ₁ / <i>c</i> (No. 14)	<i>P</i> 2 ₁ / <i>c</i> (No. 14)
<i>a</i> /Å	14.71(2)	9.381(4)
<i>b</i> /Å	3.828(4)	3.807(2)
<i>c</i> /Å	20.45(3)	16.168(8)
β /°	97.8(1)	102.40(4)
<i>V</i> /Å ³	1141(3)	563.9(5)
<i>Z</i>	4	2
<i>F</i> (000)	656	328
μ (Mo <i>K</i> _α)/cm ⁻¹	109.8	131.8
Crystal dimensions/mm ³	0.005 × 0.007 × 1.6	0.050 × 0.076 × 0.40
Scan-mode	ω -scan	ω -scan
Basic scan-width/°	1.05	2.50
Scan-speed/° min ⁻¹	0.45–0.90	0.75–1.50
2 θ range/°	4.0–100.0	2.0–100.0
Transmission factor range	–	0.1883–0.5323
Measured reflections	1403	700
Unique observed reflections [<i>I</i> ≥ 3 σ (<i>I</i>)]	486	382
Number of refined parameters	95	48
$R = \sum F_o - F_c / \sum F_o $	0.062	0.059
$R_w = (\sum w[F_o - F_c]^2 / \sum w F_o ^2)^{1/2}$	0.061	0.050

The reduction of **3** proceeds most probably via the formation of 1,2,3,4,5,6,7-heptaCN (**4**) which is consistent with the work of Brady *et al.*,¹⁷ who found that the reaction between **3** and LAH results in the formation of **4**. Brown *et al.*,¹⁸ have studied reductive dehalogenation by LAH of substituted benzenes and found that sterically more crowded halogen atoms in aromatic compounds are more easily reduced. Furthermore, electron-withdrawing substituents have been reported to enhance the reaction rate.

Thus, when **3** is reduced by LAH, one of the chlorine atoms in the α -positions are the first to be reduced. The heptaCN (**4**) formed, has three α -chlorine atoms left. However, the α -chlorine atoms in the four- and five-positions, are sterically more crowded and thus more readily reduced.

According to GLC, TLC and HPLC equipped with a RP-18 column, one major hexaCN was obtained in reduction of **3**. However, when the product was further analyzed by ¹H and ¹³C NMR spectroscopy it was found to be a mixture of two isomers. The isomers were separated on a 2-(1-pyrenyl)ethyl(dimethyl)silylated silica gel column by HPLC. Crystallographic data showed that the structures of the two isomers were 1,2,3,4,6,7-hexaCN (**5**) and 1,2,3,5,6,7-hexaCN (**6**). The two isomers were formed in about the same amount and the α -chlorine atoms in the four and five position thus seem to be equally easily reduced by LAH. Only trace amounts of other hexaCNs were formed.

The reduction of **3** proceeds via **4** to the two hexaCNs **5** and **6**, and then to several penta- and tetra-CN. It should be noted that **5** and **6** are more easily isolated if the reduction is stopped at the stage when there is no **4** remaining. The pentaCNs are easily separated from **5** and **6** while **4** partly co-elutes with **5** and **6** on the charcoal column used in the clean-up procedure.

The two hexaCNs **5** and **6** were separated from the less-chlorinated naphthalenes and trace amounts of other hexaCNs using a charcoal column (Fig. 1). Subsequently, the mixture of the two isomers were separated by HPLC using a semipreparative 2-(1-pyrenyl)ethyl(dimethyl)silylated silica gel column. The mechanisms of separation of the 2-(1-pyrenyl)ethyl(dimethyl)silylated silica phase have recently been discussed by Haglund *et al.*¹³ It is notable that no other LC method led to the successful separation of **5** and **6**. Furthermore, **5** and **6** have hitherto not been successfully separated by any GLC method.

The structures were determined by direct methods using the program SHELXS-86¹⁹ and the models obtained were refined by full-matrix least-squares calculations with the program SHELX-76.²⁰ The figures were drawn with the program PLUTO.²¹ Coefficients for the analytical approximation of the scattering factors were taken from Ref. 22. All non-hydrogen atoms could be located in the first electron density map. The hydrogen positions were calculated and the distances between the hydrogens and the corresponding carbons to which they were connected were constrained in the least-squares calculations to be 1.08 Å. The hydrogen atoms were given the same isotropic displacement factors as the carbon atoms to which they were connected. All chlorine atoms were refined anisotropically, and the rest of the atoms isotropically. The weighting scheme employed, $w = 1/[\sigma^2(F)]$, was determined from counting statistics. There were no changes larger than 0.002 σ in any of the parameters varied in the last least-squares cycle. For both compounds, atomic coordinates and equivalent isotropic displacement parameters are listed in Table 2, bond lengths in Table 3 and some selected bond angles in Table 4. The numbering scheme of the molecules is shown in Fig. 2. A list of the observed and the calculated

Table 2. Fractional atomic coordinates, estimated standard deviations given in parentheses and $U_{eq}/\text{\AA}^2$.^a

Atom	x	y	z	U_{eq}
5 (1,2,3,4,6,7-hexachloronaphthalene)				
Cl1	0.6870(4)	0.0274(21)	0.3992(3)	0.043(3)
Cl2	0.8113(5)	0.9209(22)	0.7775(3)	0.048(3)
Cl3	0.8986(4)	0.1715(21)	0.4346(3)	0.045(3)
Cl4	0.6029(5)	0.7751(22)	0.7431(3)	0.046(3)
Cl5	0.9454(4)	0.7447(20)	0.6781(3)	0.039(3)
Cl6	0.5275(4)	0.4256(21)	0.6088(3)	0.043(3)
C1	0.669(2)	0.281(7)	0.520(1)	0.028(7)
H1	0.594(2)	0.227(7)	0.507(1)	0.028(7)
C2	0.770(2)	0.728(8)	0.700(1)	0.026(7)
C3	0.857(2)	0.414(7)	0.551(1)	0.031(7)
H2	0.933(2)	0.462(7)	0.564(1)	0.031(7)
C4	0.703(2)	0.426(7)	0.581(1)	0.027(7)
C5	0.730(2)	0.214(8)	0.477(1)	0.042(8)
C6	0.831(1)	0.654(7)	0.661(1)	0.028(7)
C7	0.824(1)	0.279(7)	0.492(1)	0.025(7)
C8	0.675(1)	0.679(7)	0.687(1)	0.029(7)
C9	0.644(1)	0.513(7)	0.628(1)	0.024(7)
C10	0.798(2)	0.494(8)	0.598(1)	0.038(7)
6 (1,2,3,5,6,7-hexachloronaphthalene)				
Cl1	0.4230(4)	0.1137(14)	0.6524(3)	0.065(2)
Cl2	0.3167(4)	0.2896(13)	0.4623(2)	0.050(2)
Cl3	-0.2055(4)	0.2301(14)	0.2481(2)	0.049(2)
C1	-0.003(1)	0.177(4)	0.390(9)	0.038(4)
H1	0.072(1)	0.318(4)	0.540(9)	0.038(4)
C2	0.198(1)	0.140(4)	0.518(8)	0.044(5)
C3	0.244(2)	0.039(4)	0.602(9)	0.044(5)
C4	0.048(1)	0.076(4)	0.476(9)	0.040(4)
C5	-0.148(1)	0.110(4)	0.352(9)	0.042(5)

$$^a U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

structure factor amplitudes can be obtained on request from one of the authors (L. Eriksson).

According to the crystallographic data the two hexaCNS (**5** and **6**) are coplanar compounds, Fig. 3, which is toxicologically interesting since coplanarity is considered to be a prerequisite for toxicity similar to 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD).²³ A mixture of **5** and **6** has also been reported to induce the enzyme systems involved in this type of toxicity.¹⁰

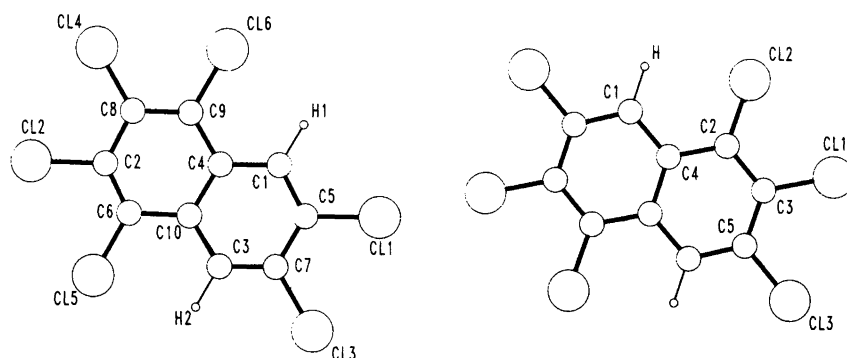


Fig. 2. Numbering scheme of (a) 1,2,3,4,6,7-hexachloronaphthalene and (b) 1,2,3,5,6,7-hexachloronaphthalene.

Table 3. Bond lengths (Å). Estimated standard deviations are given in parentheses.

5 (1,2,3,4,6,7-hexachloronaphthalene)	
C5—Cl1	1.78(0.02)
C2—Cl2	1.77(0.02)
C7—Cl3	1.76(0.02)
C8—Cl4	1.71(0.02)
C6—Cl5	1.71(0.02)
C9—Cl6	1.73(0.02)
C1—H1	1.12(0.02)
C3—H2	1.13(0.02)
C1—C4	1.40(0.03)
C1—C5	1.36(0.03)
C2—C6	1.30(0.03)
C3—C7	1.33(0.03)
C5—C7	1.40(0.03)
C2—C8	1.40(0.03)
C4—C9	1.42(0.03)
C8—C9	1.39(0.03)
C3—C10	1.41(0.03)
C4—C10	1.42(0.02)
C6—C10	1.45(0.03)
6 (1,2,3,5,6,7-hexachloronaphthalene)	
C3—Cl1	1.72(0.01)
C2—Cl2	1.69(0.01)
C5—Cl3	1.72(0.01)
C1—H	1.14(0.01)
C2—C3	1.39(0.02)
C1—C4	1.43(0.02)
C2—C4	1.44(0.02)
C4—C4	1.44(0.03)
C1—C5	1.39(0.02)
C3—C5	1.40(0.02)

Acknowledgements. We thank Professor N. Tanaka and Nacalai Tesque for the gift of a semipreparative PYE column. Dr Rolf Johansson is gratefully acknowledged for the NMR analyses and Lars Olsson for his help in preparing a large amount of octachloronaphthalene. Financial support was granted by the Swedish Environmental Protection Agency.

Table 4. Selected bond angles (°) with estimated standard deviations in parentheses.

5 (1,2,3,4,6,7-hexachloronaphthalene)	
C4-C1-H1	120(2)
C5-C1-H1	122(2)
C5-C1-C4	118(2)
C6-C2-Cl2	118(2)
C8-C2-Cl2	116(2)
C8-C2-C6	126(2)
C7-C3-H2	120(2)
C10-C3-H2	119(2)
C10-C3-C7	121(2)
C9-C4-C1	121(2)
C10-C4-C1	120(2)
C10-C4-C9	119(2)
C1-C5-C11	118(2)
C7-C5-Cl1	119(2)
C7-C5-C1	123(2)
C2-C6-Cl5	124(2)
C10-C6-Cl5	117(2)
C10-C6-C2	118(2)
C3-C7-Cl3	120(2)
C5-C7-Cl3	120(2)
C5-C7-C3	120(2)
C2-C8-Cl4	122(2)
C9-C8-Cl4	121(2)
C9-C8-C2	116(2)
C4-C9-Cl6	119(2)
C8-C9-Cl6	119(2)
C8-C9-C4	122(2)
C4-C10-C3	118(2)
C6-C10-C3	123(2)
C6-C10-C4	119(2)
6 (1,2,3,5,6,7-hexachloronaphthalene)	
C4-C1-H	121(1)
C5-C1-H	121(1)
C5-C1-C4	118(1)
C3-C2-Cl2	121(1)
C4-C2-Cl2	119(1)
C4-C2-C3	119(1)
C2-C3-Cl1	119(1)
C2-C4-C1	121(1)
C1-C5-Cl3	117(1)

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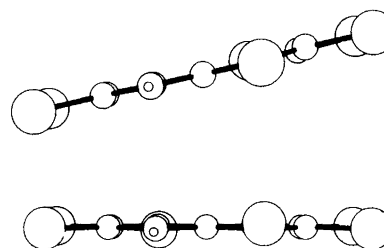


Fig. 3. Perspective view along the central C-C bond in (a) 1,2,3,4,6,7-hexachloronaphthalene and (b) 1,2,3,5,6,7-hexachloronaphthalene.

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Received July 16, 1991.