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(±)-1-*exo*-Hydroxy-4,5,6,7,8,8-hexachloro-4,7-*endo*-3a,7a-*endo*-3a,4,7,7a-tetrahydro-4,7-methanoindene

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Abstract. $C_{10}H_6Cl_6O$, tetragonal, $I4_1/a$, a = 25·023 (4), c = 8·433 (4) Å, M_r = 345·9, Z = 16, D_x = 1·79 g cm^{-3}, μ(Mo $K\alpha$) = 12·70 cm^{-1}, R = 0·044, 920 observed reflexions. The molecules are hydrogen bonded through the hydroxyl groups to form a regular spiral of O atoms down the fourfold screw axis with intramolecular O—H···O distances ranging from 2·451 to 2·703 Å. The *exo*-hydroxy configuration previously proposed on the basis of chemical evidence has been confirmed.

Introduction. The title compound is commonly known as 1-hydroxychlordene and is an inactive breakdown product of the cyclodiene insecticide heptachlor [(±)-1-*exo*-4,5,6,7,8,8-heptachloro-4,7-*endo*-3a,7a-*endo*-3a,4,-7,7a-tetrahydro-4,7-methanoindene] (Brooks, 1974). Colourless crystals were grown from aqueous isopropyl alcohol. 1884 reflexions with $I > 2{\cdot}5\sigma(I)$ were considered observed out of 2318 independent reflexions collected from one crystal (0·3 × 0·3 × 0·5 mm) mounted on a Philips PW 1100 four-circle diffractometer ($2\theta_{\max}$ = 50°, monochromatic Mo $K\alpha$ radiation). However, a reduced data block of 920 observed reflexions was used in refinement ($2\theta_{\max}$ = 37°). Standard reflexions showed no change in intensity during data collection although the crystal turned yellow. No correction was made for absorption [μ(Mo $K\alpha$) = 12·70 cm^{-1}].

The structure was solved using the centric direct-methods approach of Sheldrick (1976) and refined in space group $I4_1/a$. Full-matrix least-squares refinement with unit weights and anisotropic thermal parameters for Cl atoms gave a final R ($= \sum||F_o| - |F_c||/\sum F_o$) = 0·044. H positions were located in a difference-Fourier map and were refined with the isotropic temperature factor fixed at the value of the adjacent bonded atom. A final difference-Fourier map revealed no features larger than 0·19 e Å^{-3}. Positional and isotropic thermal parameters are given in Table 1.*

Table 1. *Atomic positional parameters* (×10^4) *and, for* C, H *and* O, *isotropic thermal parameters* (×10^3) *with estimated standard deviations in parentheses*

	x	y	z	U (Å^2)
Cl(1)	897 (1)	8075 (1)	3159 (3)	
Cl(2)	713 (1)	9110 (1)	4475 (3)	
Cl(3)	1995 (1)	8889 (1)	3057 (2)	
Cl(4)	2474 (1)	7784 (1)	4704 (3)	
Cl(5)	1528 (1)	7272 (1)	7173 (3)	
Cl(6)	454 (1)	8033 (1)	7007 (3)	
O(10)	2438 (2)	9517 (2)	7598 (7)	43 (2)
C(1)	1309 (3)	8742 (3)	7379 (10)	35 (2)
C(2)	1738 (3)	8984 (3)	6251 (10)	34 (2)
C(3)	1700 (3)	8615 (3)	4770 (9)	35 (2)
C(4)	1879 (3)	8060 (3)	5238 (9)	31 (2)
C(5)	1513 (3)	7861 (3)	6158 (9)	33 (2)
C(6)	1073 (3)	8269 (3)	6369 (9)	34 (2)
C(7)	1627 (4)	8579 (4)	8804 (11)	48 (3)
C(8)	2134 (4)	8690 (3)	8694 (10)	42 (2)
C(10)	2271 (3)	8983 (3)	7185 (10)	36 (2)
C(11)	1093 (3)	8523 (3)	4668 (9)	36 (2)
H(1)	1021 (25)	9031 (25)	7603 (78)	37
H(2)	1634 (24)	9373 (26)	5913 (76)	35
H(7)	1451 (27)	8400 (26)	9722 (90)	49
H(8)	2500 (27)	8619 (25)	9386 (81)	44
H(10)	2578 (26)	8797 (25)	6605 (78)	38
HO(10)	2590 (25)	9684 (28)	6811 (90)	44

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33658 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

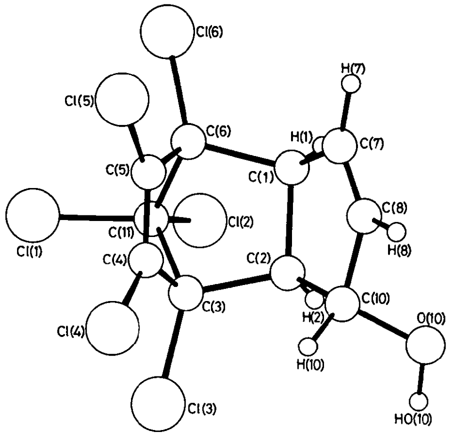

Fig. 1. The structure of 1-hydroxychlordene viewed perpendicular to the plane of C(1), C(2) and C(3).

Table 2. *Interatomic distances* (Å) *with estimated standard deviations in parentheses*

	Title compound	Heptachlor*
Cl(1)–C(11)	1·765 (8)	1·776 (5)
Cl(2)–C(11)	1·757 (8)	1·774 (5)
Cl(3)–C(3)	1·761 (8)	1·748 (5)
Cl(4)–C(4)	1·702 (8)	1·710 (5)
Cl(5)–C(5)	1·705 (8)	1·700 (6)
Cl(6)–C(6)	1·742 (8)	1·765 (6)
O(10)–C(10)	1·443 (9)	–
O(10)–HO(10)	0·87 (7)	–
C(1)–C(2)	1·557 (11)	1·573 (7)
C(1)–C(6)	1·573 (11)	1·560 (7)
C(1)–C(7)	1·499 (13)	1·498 (8)
C(1)–H(1)	1·04 (6)	1·13
C(2)–C(3)	1·556 (11)	1·554 (7)
C(2)–C(10)	1·549 (11)	1·572 (7)
C(2)–H(2)	1·05 (7)	0·97
C(3)–C(4)	1·512 (11)	1·519 (8)
C(3)–C(11)	1·539 (11)	1·560 (7)
C(4)–C(5)	1·299 (11)	1·329 (8)
C(5)–C(6)	1·512 (11)	1·522 (7)
C(6)–C(11)	1·570 (11)	1·562 (7)
C(7)–C(8)	1·302 (14)	1·324 (8)
C(7)–H(7)	1·00 (7)	0·80
C(8)–C(10)	1·508 (12)	1·484 (8)
C(8)–H(8)	1·10 (7)	0·82
C(10)–H(10)	1·02 (7)	0·88
HO(10)···O(10′)	1·86 (7)	

* Shields & Kennard (1973).

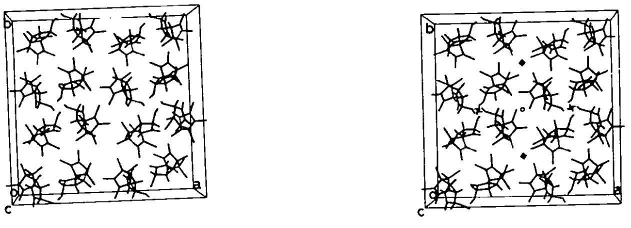

Fig. 2. Stereoscopic view of the packing of the title compound viewed down the *c* axis.

Table 3. *Interatomic angles* (°) *with estimated standard deviations in parentheses*

	Title compound	Heptachlor*
C(2)–C(1)–C(6)	102·7 (4)	102·2 (3)
C(2)–C(1)–C(7)	103·3 (4)	103·8 (3)
C(6)–C(1)–C(7)	115·4 (4)	114·8 (3)
C(1)–C(2)–C(3)	102·6 (4)	103·4 (3)
C(1)–C(2)–C(10)	106·4 (4)	105·8 (3)
C(3)–C(2)–C(10)	117·4 (4)	114·9 (3)
C(2)–C(3)–C(4)	108·5 (4)	107·7 (3)
C(2)–C(3)–C(11)	101·2 (4)	100·6 (3)
C(2)–C(3)–Cl(3)	113·7 (4)	115·1 (3)
C(4)–C(3)–C(11)	99·8 (4)	98·6 (3)
C(4)–C(3)–Cl(3)	116·6 (4)	115·9 (3)
C(11)–C(3)–Cl(3)	115·2 (4)	116·7 (3)
Cl(4)–C(4)–C(3)	124·3 (4)	124·5 (3)
Cl(4)–C(4)–C(5)	128·2 (4)	126·8 (3)
C(3)–C(4)–C(5)	107·4 (5)	108·4 (3)
Cl(5)–C(5)–C(4)	128·0 (4)	128·5 (3)
Cl(5)–C(5)–C(6)	122·7 (4)	123·7 (3)
C(4)–C(5)–C(6)	108·9 (5)	107·3 (3)
C(1)–C(6)–C(5)	107·3 (4)	108·0 (3)
C(1)–C(6)–C(11)	100·1 (4)	100·8 (3)
C(1)–C(6)–Cl(6)	114·9 (4)	114·5 (2)
C(5)–C(6)–C(11)	98·2 (4)	99·2 (3)
C(5)–C(6)–Cl(6)	117·0 (4)	115·7 (3)
C(11)–C(6)–Cl(6)	116·6 (4)	116·6 (3)
C(1)–C(7)–C(8)	113·7 (6)	112·6 (3)
C(7)–C(8)–C(10)	112·7 (5)	114·2 (3)
C(2)–C(10)–C(8)	103·5 (4)	103·6 (3)
C(2)–C(10)–O(10)	111·8 (6)	110·8 (3)†
C(8)–C(10)–O(10)	108·2 (5)	110·2 (3)†
Cl(1)–C(11)–C(6)	113·1 (4)	113·8 (3)
Cl(1)–C(11)–Cl(2)	108·3 (4)	107·5 (2)
Cl(2)–C(11)–C(3)	114·5 (4)	114·3 (3)
Cl(2)–C(11)–C(6)	113·9 (4)	114·7 (3)
C(3)–C(11)–C(6)	92·3 (4)	92·9 (3)
C(3)–C(11)–Cl(2)	114·5 (4)	114·3 (3)
C(6)–C(11)–Cl(2)	113·9 (4)	114·7 (3)
O(10)–HO(10)···O(10′)	170·4	–

* Shields & Kennard (1973).
† Angles involving Cl(7).

Discussion. The structure determination of 1-hydroxychlordene confirms the 1-*exo*-hydroxy configuration for the molecule (Fig. 1) proposed previously on the basis of chemical evidence (Brooks, 1974). The numbering scheme employed conforms to the convention used for a number of cyclodiene insecticides determined in this laboratory (see Smith & Kennard, 1977). The *exo* configuration is the same as that found for the equivalent Cl in heptachlor (Shields & Kennard, 1973). In fact, the structure is very similar to heptachlor and a comparison has been made of bond distances and angles (Tables 2 and 3).

The 1-hydroxychlordene molecule is dissymmetric but crystallizes in a centrosymmetric space group; consequently, both optical forms are present. Unlike heptachlor, the molecule contains a hydroxyl O which results in an intricate hydrogen-bonding system unique among the reported structures of cyclodiene analogues. The hydrogen bonds, involving intermolecular O—H···O distances ranging from 2·451 to 2·703 Å, form a spiral of molecules down the fourfold screw axes (Fig. 2). The O(10)—HO(10)···O(10′) angle is 170·40° while the HO(10)···O(10′) distance is 1·860 Å. The molecules radiating out from the 4_1 or 4_3 axes at $\frac{1}{4},0,z$; $\frac{1}{4},\frac{1}{2},z$; $\frac{3}{4},0,z$; and $\frac{3}{4},\frac{1}{2},z$ interlock with other molecules such that substituted Cl atoms from different molecules are kept apart. They are arranged in distorted tetrahedra (interatomic distances 2·58 and 3·90 Å) around $\bar{4}$ at $0,\frac{1}{4},\frac{1}{8}$; $0,\frac{3}{4},\frac{7}{8}$; $\frac{1}{2},\frac{3}{4},\frac{5}{8}$; $\frac{1}{2},\frac{1}{4},\frac{3}{8}$. There are also centres of inversion at 0,0,0; $0,\frac{1}{2},0$; $\frac{1}{4},\frac{1}{4},\frac{1}{4}$; $\frac{3}{4},\frac{1}{4},\frac{1}{4}$; $\frac{1}{2},\frac{1}{2},\frac{1}{2}$; $\frac{1}{2},0,\frac{1}{2}$; $\frac{3}{4},\frac{3}{4},\frac{3}{4}$; and $\frac{1}{4},\frac{3}{4},\frac{3}{4}$.

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2-Naphthyloxyacetic Acid

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Abstract. $C_{12}H_{10}O_3$, $M_r = 202$, monoclinic, $P2_1/c$, $a = 12·298$ (5), $b = 6·830$ (3), $c = 13·407$ (7) Å, $\beta = 118·590$ (8)°, $V = 988·8$ Å^3, $Z = 4$, $D_m = 1·37$ (1), $D_c = 1·356$ g cm^{-3}. The structure, which was refined to an R of 0·083 for 1623 counter reflections, is stabilized by O—H···O hydrogen bonds and forms a centrosymmetric dimer.

Introduction. The study of 2-naphthyloxyacetic acid (BNAA) forms part of a project on structure and function of plant growth hormones. BNAA, a synthetic auxin, has two effects on growth, one relating to cell elongation and the other to the rate of cell division. Auxins possess a naphthalene or benzene group and a side chain of two or more methylene C atoms. Information regarding the orientation of the side chain with respect to the naphthalene group is of interest.

Pink transparent parallelepipeds were obtained from a solution in ethanol. The space group was determined from systematic absences observed on Weissenberg photographs. Cell parameters were obtained by a least-squares fit of 25 θ values measured on a four-circle Picker diffractometer with Cu $K\alpha$ radiation ($\lambda = 1·54178$ Å). Intensities of 1623 reflections with $2\theta \leq 130°$ were collected with a crystal mounted about b, Ni-filtered Cu $K\alpha$ radiation, a θ–2θ scan, and a 2° min^{-1} scan speed. The scan range was 2° and background was measured on either side of the peak for 10 s. 60 reflections had $I < 2\sigma(I)$ and were excluded. The data were corrected for Lorentz and polarization factors but not for absorption.

The structure was solved with *MULTAN* (Main, Woolfson & Germain, 1971). Improved $|E|$ values were calculated by correcting for molecular scattering with the Debye (1915) formula for the naphthalene group. The E map computed with a set of phases having the fourth highest figure of merit (1·988) revealed the positions of eight atoms, the remaining

Table 1. *Positional parameters*

	x	y	z
C(1)	0·4467 (4)	0·7624 (6)	0·0355 (4)
C(2)	0·4013 (4)	0·5776 (6)	0·0666 (3)
C(3)	0·2685 (4)	0·3082 (6)	−0·0241 (3)
C(4)	0·1835 (4)	0·2211 (7)	−0·1286 (3)
C(5)	0·1232 (3)	0·0542 (6)	−0·1302 (3)
C(6)	0·1452 (4)	−0·0381 (6)	−0·0271 (3)
C(7)	0·0855 (4)	−0·2130 (7)	−0·0249 (4)
C(8)	0·1075 (5)	−0·2953 (8)	0·0765 (4)
C(9)	0·1917 (4)	−0·2069 (7)	0·1796 (4)
C(10)	0·2520 (4)	−0·0394 (6)	0·1806 (3)
C(11)	0·2301 (4)	0·0506 (6)	0·0774 (3)
C(12)	0·2919 (4)	0·2250 (6)	0·0775 (3)
O(1)	0·3243 (3)	0·4759 (4)	−0·0348 (2)
O(2)	0·5195 (3)	0·8635 (4)	0·1251 (2)
O(3)	0·4178 (3)	0·8072 (4)	−0·0615 (2)

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* Contribution No. 495.