anti-1,6,7,8,9,14,15,16-octachloropentacyclo[12.2.1.$1^{6,9}.0^{2,13}.0^{5,1}$]octadeca-7,15-diene (4) (Garcia, Fronczek & McLauglin, 1991*b*) [C(5)—C(6) 1.587 (3), C(5′)—C(6′) 1.587 (3) Å], *endo-endo-anti*-1,6,7,8,9,14,15,16-octachloro-17,17,18,18-tetramethoxypentacyclo[12.2.1.$1^{6,9}.0^{2,13}.0^{5,10}$]octadeca-7,15-diene (5) (Garcia, Fronczek & McLauglin, 1991*c*) [C(5)—C(6) 1.579 (3), C(5′)—C(6′) 1.579 (3) Å], *endo-endo-anti*-17,17,18,18-tetramethoxypentacyclo[12.2.1.$1^{6,9}.0^{2,13}.0^{5,10}$]octadeca-7,15-diene (6) (Garcia *et al.*, 1991*c*) [C(5)—C(6) 1.575 (1), C(5′)—C(6′) 1.575 (1) Å] and *endo-endo-anti*-pentacyclo[12.2.1.$1^{6,9}.0^{2,13}.0^{5,10}$]octadeca-7,15-diene-17,18-dione (7) (Garcia, Fronczek & McLauglin, 1991*d*) [C(5)—C(6) 1.576 (1), C(5′)—C(6′) 1.576 (1) Å]. Bond lengths C(12)—C(13) 1.318 (5), C(20)—C(21) 1.312 (5) Å of the title compound are similar to those of (2) [C(12*a*)—C(13*a*) 1.317 (5) and C(12*a*′)—C(13*a*′) 1.317 (5), C(12*b*)—C(13*b*) 1.314 (5) Å respectively for the two independent molecules], (3) [C(12)—C(13) 1.317 (4), C(12′)—C(13′) 1.314 (4) Å], (4) [C(12)—C(13) 1.327 (3) and C(12′)—C(13′) 1.327 (3) Å], (5) [C(12)—C(13) 1.324 (3) and C(12′)—C(13′) 1.324 (3) Å], (6) [C(12)—C(13) 1.329 (1) and C(12′)—C(13′) 1.329 (1) Å] and (7) [C(12)—C(13) 1.325 (2) and C(12′)—C(13′) 1.325 (2) Å]. Bond angles at the geminal chlorine bridge of the title compound [C(11)—C(18)—C(14) 92.0 (2) and C(19)—C(23)—C(22) 92.5 (2)°] are similar to those of (2) [92.3 (2), 92.5 (2) and 92.3 (2), 92.5 (2)° respectively from the two independent molecules] and (3) [92.3 (2) and 92.4 (2)°]. The bond angle at the geminal methylene bridge C(9)—C(17)—C(16) 94.6 (3)° is similar to that of (4) [92.2 (2)°].

Torsion angles C(8)—C(1)—C(2)—C(3) and C(4)—C(5)—C(6)—C(7) at the ring fusion of the title compound are −6.7 (4) and −3.9 (4)° in accordance with those of (2) [±0.5 (4) and ±0.7 (4)° respectively for the two independent molecules], (3) [−0.8 (3) and 0.7 (2)°], (4) [± 0.3 (3)°], (5) [± 1.8 (3)°], (6) [±0.9 (1)°] and (7) [±0.8 (1)°].

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The Major Diels–Alder Adduct of 1,2,3,4-Tetrachloro-1,3-cyclopentadiene with 1,9,10,11-Tetrachloro-12,12-dimethoxytricyclo[8.2.1.$0^{2,8}$]dodeca-5,10-diene

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Abstract. *endo-endo-anti*-1,6,7,8,9,14,15,16-Octachloro-17,17-dimethoxypentacyclo[12.2.1.$1^{6,9}.0^{2,13}.0^{5,10}$]octadeca-7,15-diene, $C_{20}H_{20}Cl_8O_2$, $M_r = 576.0$, monoclinic, $P2_1/c$, $a = 20.078$ (2), $b = 11.4098$ (7), $c = 10.7579$ (6) Å, $\beta = 103.629$ (7)°, $V = 2395.0$ (3) Å^3, $Z = 4$, $D_x = 1.604$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 90.11$ cm^{-1}, $F(000) = 1168$, $T = 296$ K, $R = 0.051$ for 3061 observations with $I > 3\sigma(I)$ (of 4911 unique

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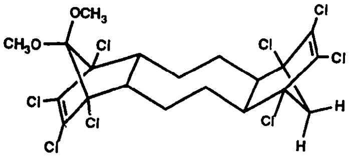
data). The cyclooctane ring adopts the chair conformation. The zero torsion angles of the chair occur at the ring-fusion bonds, with magnitudes of 2.5 (5) and 1.4 (5)°. The endocyclic torsion angles about the bonds adjacent to the eclipsed bond vary in magnitude from 75.2 (2) to 78.2 (4)°, and the torsion angles about the bonds comprising the sides of the chair are −119.5 (4) and 116.9 (4)°. The C═C bonds have lengths of 1.329 (5) and 1.322 (5) Å.

Experimental. The title compound (1) was prepared by slowly heating a 3:1 mixture of 1,2,3,4-tetrachloro-1,3-cyclopentadiene (Roedig & Hörnig, 1955) with 1,9,10,11-tetrachloro-12,12-dimethoxytricyclo[8.2.1.$0^{2,8}$]dodeca-5,10-diene (Akhtar, Fray & Yarrow, 1968) to 406–413 K for 3 h and filtering the white solid. Crystals grown by slow cooling of toluene, m.p. > 578 K (dec.), were suitable; a thin colorless plate with dimensions 0.07 × 0.15 × 0.30 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $22 > \theta > 17°$. The ω–2θ scans were designed for $I = 25\sigma(I)$, subject to maximum scan time = 120 s, scan rates varied from 0.63 to 3.30° min^{-1}. One quadrant of data having $2 < \theta < 75°$, $0 \leq h \leq 25$, $0 \leq k \leq 14$, $-13 \leq l \leq 13$ was measured and corrected for background, Lorentz, polarization and absorption. Absorption corrections were based on ψ scans, with minimum relative transmission coefficient 0.476. Three standard reflections (400, 020, 004), measured every 10 000 s, decayed in intensity 7.9, 2.0 and 18.9%, respectively, and an anisotropic decay correction was applied. The space group was determined by systematic absences $h0l$ with l odd and $0k0$ with k odd. Redundant $0kl$ and $0k\bar{l}$ data were averaged; $R_{\rm int} = 0.020$. The structure is isomorphous with the analogous compound having two Cl substituents at C(13) (Fronczek, Garcia & McLaughlin, 1991) and coordinates from that structure were used as a beginning model. The methoxy groups were then located by ΔF synthesis. Refinement was carried out by full-matrix least squares based on F, using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$, using the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974) and anomalous coefficients of Cromer (1974). Heavy-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF synthesis and placed in calculated positions with C—H 0.95 Å and $B = 1.3$ times the $B_{\rm eq}$ value for the C atom. Final $R = 0.051$ for 3061 observed data (0.103 for all 4911 data), $wR = 0.059$ and $S = 2.347$ for 272 variables. Maximum shift was 0.09σ in the final cycle, maximum residual density 0.57 and minimum −0.35 e Å^{-3}. An extinction coefficent was refined to $g = 3.7\,(4) \times 10^{-7}$, where the correction factor $(1 + gIc)^{-1}$ was applied to F_c. The fractional coordinates of the title compound are given in Table 1. Fig. 1 is a perspective drawing showing the atom numbering for the title molecule, and Fig. 2 shows the unit cell. Bond distances, angles and selected torsion angles are presented in Table 2.*



(1)

Table 1. *Coordinates and equivalent isotropic thermal parameters*

$B_{\rm eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$B_{\rm eq}$ (Å^2)
Cl(1)	0.72332 (6)	0.1841 (1)	1.0916 (1)	4.97 (3)
Cl(2)	0.72664 (7)	−0.1003 (1)	1.1326 (1)	5.65 (3)
Cl(3)	0.62158 (7)	−0.2312 (1)	0.8766 (1)	5.82 (3)
Cl(4)	0.55152 (6)	−0.0254 (1)	0.6673 (1)	6.08 (3)
Cl(5)	0.85403 (7)	−0.1952 (1)	0.4182 (1)	5.13 (3)
Cl(6)	0.83728 (6)	0.0914 (1)	0.3641 (1)	4.76 (3)
Cl(7)	0.93949 (6)	0.2359 (1)	0.6125 (1)	4.45 (3)
Cl(8)	1.02417 (5)	0.0388 (1)	0.8198 (1)	4.59 (3)
O(1)	0.6141 (2)	0.2137 (3)	0.7968 (3)	5.02 (8)
O(2)	0.5740 (2)	0.1148 (3)	0.9534 (3)	6.06 (9)
C(1)	0.7373 (2)	0.0943 (4)	0.8580 (3)	3.26 (9)
C(2)	0.6889 (2)	0.0319 (4)	0.7395 (4)	3.50 (9)
C(3)	0.7158 (2)	−0.0762 (4)	0.6841 (4)	3.8 (1)
C(4)	0.7656 (2)	−0.0487 (4)	0.5983 (4)	3.6 (1)
C(5)	0.8381 (2)	−0.0954 (4)	0.6469 (4)	3.34 (9)
C(6)	0.8871 (2)	−0.0293 (4)	0.7623 (3)	3.12 (9)
C(7)	0.8580 (2)	0.0757 (4)	0.8197 (4)	3.41 (9)
C(8)	0.8093 (2)	0.0448 (4)	0.9057 (4)	3.36 (9)
C(9)	0.6936 (2)	0.0904 (4)	0.9593 (4)	3.57 (9)
C(10)	0.6847 (2)	−0.0365 (4)	0.9943 (4)	3.7 (1)
C(11)	0.6430 (2)	−0.0863 (4)	0.8938 (4)	4.0 (1)
C(12)	0.6242 (2)	0.0054 (4)	0.7906 (4)	3.8 (1)
C(13)	0.6204 (2)	0.1157 (4)	0.8745 (4)	4.1 (1)
C(14)	0.8814 (2)	−0.0928 (4)	0.5433 (4)	3.37 (9)
C(15)	0.8851 (2)	0.0341 (4)	0.5019 (4)	3.43 (9)
C(16)	0.9257 (2)	0.0893 (4)	0.5988 (3)	3.15 (9)
C(17)	0.9489 (2)	0.0002 (4)	0.7044 (4)	3.29 (9)
C(18)	0.9533 (2)	−0.1094 (4)	0.6259 (4)	3.8 (1)
C(19)	0.6177 (3)	0.3261 (6)	0.8515 (6)	8.9 (2)
C(20)	0.5051 (3)	0.1334 (8)	0.9023 (7)	10.8 (2)

Related literature. The single-bond distances C(2)—C(12) 1.556 (6) and C(1)—C(9) 1.553 (6) Å in the title compound are similar to analogous bond distances in 1,6,7,8,9,14,15,16-octachloro-17,17,18,18-tetramethoxypentacyclo[12.2.1.$1^{6,9}$.$0^{2,13}$.$0^{5,10}$]octadeca-7,15-diene (2) (Garcia, Fronczek & McLaughlin, 1991*a*) [1.561 (3) and 1.555 (4) Å], 17,17,18,18-tetramethoxypentacyclo[12.2.1.$1^{6,9}$.$0^{2,13}$.$0^{5,10}$]octade-

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* Lists of H-atom coordinates and thermal parameters, anisotropic thermal parameters, structure-factor amplitudes, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54453 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

ca-7,15-diene (3) (Garcia, Fronczek & McLaughlin, 1991*a*) [1.566 (1) and 1.564 (1) Å] and *endo-endo-anti*-1,6,7,8,9,14,15,16-octachloropentacyclo[12.2.1.-$1^{6,9}.0^{2,13}.0^{5,10}$]octadeca-7,15-diene (4) (Garcia, Fronczek & McLaughlin, 1991*b*) [1.553 (4) and 1.560 (3) Å]. The bond angle C(14)—C(18)—C(17) [92.9 (3)°] of the title compound is similar to the analogous bond angles of (2) [90.9 (2)°], (3) [93.9 (8)°] and (4) [92.2 (2)°]. The near-zero torsion angles C(3)—C(2)—C(1)—C(8) [2.5 (5)°] and C(4)—C(5)—C(6)—C(7) [1.4 (5)°] as well as torsion angles C(2)—C(3)—C(4)—C(5) [116.9 (4)°] and C(1)—C(8)—C(7)—C(6) [−119.5 (4)°] in the title compound are in accord with (2) [±1.8 (3) and ±119.0 (2)°, respectively], (3) [±0.9 (1) and ±117.8 (1)°, respectively] and (4) [±0.3 (3) and ±117.8 (2)°, respectively].

The C(9)—C(13)—C(12) bond angle [91.2 (3)°] of the title compound is similar to the analogous bond angles of (2) and (3) [90.9 (2) and 93.88 (8)° respectively]. Torsion angles O(1)—C(13)—O(2)—C(20) and O(2)—C(13)—O(1)—C(19) of the title molecule [50.6 (7) and 54.3 (6)° respectively] are in accord with those of (2) [52.2 (3) and 56.5 (3)° respectively].

We thank the Louisiana Educational Quality Support Fund Grant No. LEQSF(1987-90)-RD-A-5 for support of this research.

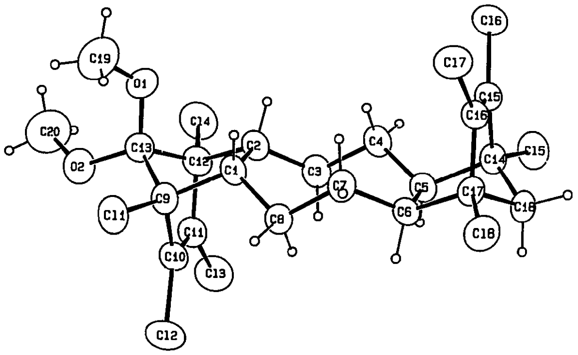

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the title molecule, representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radius.

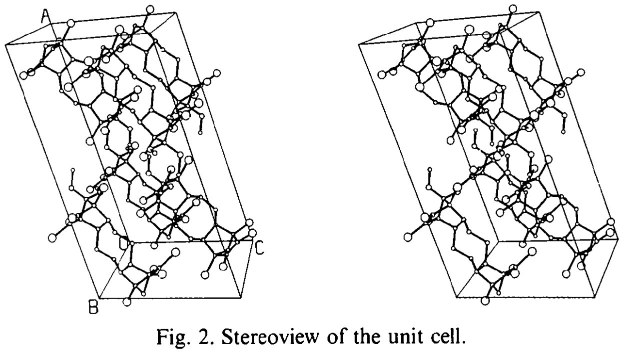

Fig. 2. Stereoview of the unit cell.

Table 2. *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

Cl(1)—C(9)	1.768 (4)	C(3)—C(4)	1.543 (6)
Cl(2)—C(10)	1.692 (4)	C(4)—C(5)	1.522 (5)
Cl(3)—C(11)	1.707 (5)	C(5)—C(6)	1.582 (5)
Cl(4)—C(12)	1.761 (4)	C(5)—C(14)	1.568 (6)
Cl(5)—C(14)	1.769 (4)	C(6)—C(7)	1.525 (6)
Cl(6)—C(15)	1.696 (4)	C(6)—C(17)	1.553 (6)
Cl(7)—C(16)	1.697 (4)	C(7)—C(8)	1.538 (6)
Cl(8)—C(17)	1.770 (4)	C(9)—C(10)	1.517 (6)
O(1)—C(13)	1.383 (6)	C(9)—C(13)	1.564 (5)
O(1)—C(19)	1.406 (8)	C(10)—C(11)	1.329 (5)
O(2)—C(13)	1.401 (6)	C(11)—C(12)	1.507 (6)
O(2)—C(20)	1.378 (6)	C(12)—C(13)	1.561 (7)
C(1)—C(2)	1.578 (5)	C(14)—C(15)	1.521 (6)
C(1)—C(8)	1.524 (5)	C(14)—C(18)	1.518 (5)
C(1)—C(9)	1.553 (6)	C(15)—C(16)	1.322 (5)
C(2)—C(3)	1.523 (6)	C(16)—C(17)	1.514 (5)
C(2)—C(12)	1.556 (6)	C(17)—C(18)	1.523 (6)

C(13)—O(1)—C(19)	119.7 (4)	Cl(2)—C(10)—C(11)	128.3 (4)
C(13)—O(2)—C(20)	120.3 (4)	C(9)—C(10)—C(11)	107.1 (3)
C(2)—C(1)—C(8)	117.3 (3)	Cl(3)—C(11)—C(10)	126.6 (3)
C(2)—C(1)—C(9)	102.2 (3)	Cl(3)—C(11)—C(12)	125.4 (3)
C(8)—C(1)—C(9)	113.6 (3)	C(10)—C(11)—C(12)	107.7 (4)
C(1)—C(2)—C(3)	118.3 (3)	Cl(4)—C(12)—C(2)	112.7 (3)
C(1)—C(2)—C(12)	102.0 (3)	Cl(4)—C(12)—C(11)	115.9 (3)
C(3)—C(2)—C(12)	112.6 (4)	Cl(4)—C(12)—C(13)	116.9 (3)
C(2)—C(3)—C(4)	114.2 (4)	C(2)—C(12)—C(11)	108.0 (3)
C(3)—C(4)—C(5)	115.1 (3)	C(2)—C(12)—C(13)	101.9 (3)
C(4)—C(5)—C(6)	118.1 (3)	C(11)—C(12)—C(13)	99.9 (3)
C(4)—C(5)—C(14)	113.2 (3)	O(1)—C(13)—O(2)	113.1 (4)
C(6)—C(5)—C(14)	101.7 (3)	O(1)—C(13)—C(9)	116.2 (4)
C(5)—C(6)—C(7)	117.8 (3)	O(1)—C(13)—C(12)	108.2 (3)
C(5)—C(6)—C(17)	101.3 (3)	O(2)—C(13)—C(9)	108.2 (3)
C(7)—C(6)—C(17)	114.3 (3)	O(2)—C(13)—C(12)	118.3 (4)
C(6)—C(7)—C(8)	114.9 (4)	C(9)—C(13)—C(12)	91.2 (3)
C(1)—C(8)—C(7)	113.6 (3)	Cl(5)—C(14)—C(5)	113.8 (3)
Cl(1)—C(9)—C(1)	114.1 (3)	Cl(5)—C(14)—C(15)	115.7 (3)
Cl(1)—C(9)—C(10)	114.5 (3)	Cl(5)—C(14)—C(18)	116.4 (3)
Cl(1)—C(9)—C(13)	116.8 (3)	C(5)—C(14)—C(15)	107.4 (3)
C(1)—C(9)—C(10)	108.7 (4)	C(5)—C(14)—C(18)	101.0 (3)
C(1)—C(9)—C(13)	101.2 (3)	C(15)—C(14)—C(18)	100.7 (3)
C(10)—C(9)—C(13)	99.9 (3)	Cl(6)—C(15)—C(14)	124.5 (3)
Cl(2)—C(10)—C(9)	124.5 (3)	Cl(6)—C(15)—C(16)	128.5 (3)
C(14)—C(15)—C(16)	106.6 (3)	Cl(8)—C(17)—C(18)	116.2 (3)
Cl(7)—C(16)—C(15)	126.6 (3)	C(6)—C(17)—C(16)	108.2 (3)
Cl(7)—C(16)—C(17)	125.6 (3)	C(6)—C(17)—C(18)	101.6 (3)
C(15)—C(16)—C(17)	107.2 (4)	C(16)—C(17)—C(18)	100.6 (3)
Cl(8)—C(17)—C(6)	113.8 (3)	C(14)—C(18)—C(17)	92.9 (3)
Cl(8)—C(17)—C(16)	114.9 (3)		

C(19)—O(1)—C(13)—O(2)	54.3 (6)	C(20)—O(2)—C(13)—O(1)	50.6 (7)
C(8)—C(1)—C(2)—C(3)	2.5 (5)	C(4)—C(5)—C(6)—C(7)	1.4 (5)
C(6)—C(7)—C(8)—C(1)	− 119.5 (4)	C(2)—C(3)—C(4)—C(5)	116.9 (4)

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