C2—C3—C4	110 (1)	N21—C4—C5	125 (1)
C3—C2—C1	105 (1)	N21—C4—C3	108 (1)
N21—C1—C2	111 (1)	C5—C51—C56	122 (1)
C2—C1—C20	123 (1)	C5—C51—C52	119 (1)
N21—C1—C20	125 (1)	C52—C51—C56	119 (1)
C1—C20—C201	119 (1)	C51—C52—C53	120 (1)
C1—C20—C19	126 (1)	C52—C53—C54	121 (1)
C19—C20—C201	116 (1)	C53—C54—C55	120 (1)
N24—C19—C20	125 (1)	C54—C55—C56	119 (1)
C20—C19—C18	128 (1)	C51—C56—C55	122 (1)
N24—C19—C18	107 (1)	C10—C101—C10	121 (1)
C19—C18—C17	107 (1)	C102—C101—C10	118 (1)
C18—C17—C16	109 (1)	C101—C102—C10	121 (1)
N24—C16—C17	106 (1)	C102—C103—C104	120 (1)
C17—C16—C15	126 (1)	C103—C104—C105	120 (1)
N24—C16—C15	128 (1)	C104—C105—C106	121 (1)
C16—C15—C151	116 (1)	C101—C106—C105	121 (1)
C16—C15—C14	124 (1)	C20—C201—C206	119 (1)
C14—C15—C151	120 (1)	C20—C201—C202	122 (1)
N23—C14—C15	126 (1)	C202—C201—C206	119 (1)
C15—C14—C13	124 (1)	C201—C202—C203	120 (1)
N23—C14—C13	110 (1)	C202—C203—C204	122 (1)
C14—C13—C12	105 (1)	C203—C204—C205	119 (1)
C13—C12—C11	109 (1)	C204—C205—C206	120 (1)
N23—C11—C12	109 (3)	C201—C206—C205	120 (1)
C12—C11—C10	126 (1)	C155—C156—C151	118 (1)
N23—C11—C10	124 (1)	C156—C155—C154	122 (2)
C11—C10—C101	119 (1)	C155—C154—C153	119 (2)
C11—C10—C9	123 (1)	C154—C153—C152	120 (1)
C9—C10—C101	118 (1)	C153—C152—C151	122 (1)
N22—C9—C10	127 (1)	C156—C151—C152	118 (1)
C10—C9—C8	128 (1)	C15—C151—C152	123 (1)
N22—C9—C8	105 (1)	C15—C151—C156	118 (1)

N21—C1—C20—C201	170 (1)	O31—N31—C3—C2	40 (2)
N22—C6—C5—C51	−172 (1)	O32—N31—C3—C4	36 (2)
N23—C11—C10—C101	158 (1)	O121—N121—C12—C11	−28 (2)
N24—C16—C15—C151	178 (1)	O122—N121—C12—C13	−38 (2)
N31—C3—C4—N21	−167 (1)	C51—C5—C4—N21	−157 (1)
N31—C3—C2—C1	168 (1)	C101—C10—C9—N22	174 (1)
N121—C12—C13—C14	−164 (1)	C151—C15—C14—N23	−165 (1)
N121—C12—C11—N23	161 (1)	C201—C20—C19—N24	175 (1)

The data were collected with a scan width of $(0.90 + 2.70\tan\theta)^\circ$ in ω, a scan rate of 5.5° min^{-1} and a background count for one quarter of the total time spent on each count. The structure was solved using *SHELXS*86 (Sheldrick, 1985) and refined by full-matrix least squares with *SHELX*76 (Sheldrick, 1976). All H atoms apart from H202 and the solvent H atoms were located in the difference map and refined. The high final R is attributed to the small size and poor quality of the crystal specimen.

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Lists of structure factors, anisotropic displacement parameters, least-squares-planes data, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71522 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1041]

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The Structures of *endo*-13-*syn*-Methyltricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-13-*anti*-ol, *endo*-13-*syn*-Methyltricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-13-*anti*-yl Acetate and its Corresponding *endo-endo-anti* Diels–Alder Adducts with 1,2,3,4-Tetrachloro-5,5-dimethoxy-1,3-cyclopentadiene and Hexachlorocyclopentadiene

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Abstract

endo-13-*syn*-Methyltricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-13-*anti*-ol, (1): there are four independent molecules in the asymmetric unit. In two the cyclooctene ring adopts the twist-boat conformation and in the other two, the chair conformation. There are near-

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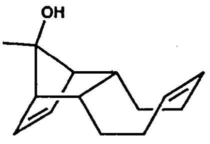
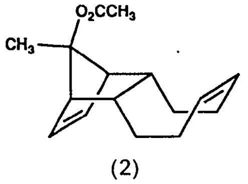
zero torsion angles at the ring-fusion bonds in the boat and chair forms [−1.1 (4), 1.6 (4), 2.2 (4) and −1.9 (4)°, respectively]. The torsion angles about the bonds comprising the sides of the twist boat are 25.7 (5) and 6.2 (6)°, and −1.0 (4) and −14.1 (5)°, respectively. The torsion angles about the bonds comprising the sides of the chair are 106.3 (4) and −109.6 (4)°, and 109.0 (3) and −107.3 (4)°, respectively. The cyclooctene and norbornene C=C bonds have lengths of 1.342 (5) and 1.307 (4) Å, 1.312 (5) and 1.313 (5) Å, 1.315 (5) and 1.308 (4) Å and 1.303 (6) and 1.305 (5) Å, respectively. *endo*-13-*syn*-Methyltricyclo[8.2.1.$0^{2,9}$]trideca-5,11-dien-13-*anti*-yl acetate (2): the cyclooctene ring adopts the twist-boat conformation. There is a near-zero torsion angle of the boat at the ring fusion bond [−0.1 (3)°]. The torsion angles about the bonds comprising the sides of the boat are −10.0 (3) and −27.8 (3)°. The cyclooctene and norbornene C=C bonds have lengths of 1.296 (2) and 1.314 (5) Å, respectively. *endo,endo,anti*-18,18-Dimethoxy-17-*syn*-methyl-6,7,8,9-tetrachloropentacyclo[12.2.1.$1^{6,9}$.$0^{2,13}$.$0^{5,10}$]octadeca-7,15-dien-17-*anti*-yl acetate (3) and *endo,endo,anti*-6,7,8,9,18,18-Hexachloro-17-*syn*-methylpentacyclo [12.2.1.$1^{6,9}$.$0^{2,13}$.$0^{5,10}$]octadeca-7,15-dien-17-*anti*-yl acetate (4): the cyclooctane ring adopts the chair conformation. The near-zero torsion angles of the chair occur at the ring fusion bonds [−1.7 (4) and −1.4 (4) for (3) and −3.3 (2) and −2.7 (2)° for (4)]. The torsion angles about the bonds comprising the sides of the chair are 119.1 (3) and −115.9 (3)° for (3) and 119.4 (2) and −116.2 (2)° for (4). The HC=CH and ClC=CCl bonds are of lengths 1.317 (5) and 1.322 (5) Å for (3), and 1.312 (3) and 1.319 (3) Å for (4), respectively.

Comment

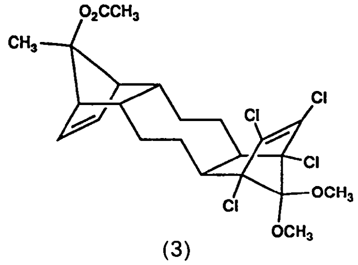
The crystal structures of a number of substituted cycloocta(e)nes have been determined in our laboratory over the past two years (Garcia, Fronczek & McLaughlin, 1991*a*,*b*,*c*,*d*, 1992*a*,*b*, 1994; Li, Fronczek & McLaughlin, 1992) on account of a study of conformation-dependent π–σ–π electronic interactions (Garcia, Fronczek & McLaughlin, 1991*e*; Garcia & McLaughlin, 1991). The crystal structure determinations of the title compounds are part of an ongoing program of structure analysis of some new derivatives bearing the cycloocta(e)ne moiety. Crystals suitable for analysis of (1) (Eaton, Sidhu, Langford, Cullison & Pietruszewski, 1981), m.p. 330 K, were deposited in the reaction flask; crystals of (2) (Eaton, Sidhu, Langford, Cullison & Pietruszewski, 1981), m.p. 345–346 K, were grown by slow evaporation of a 1:1:10 tetrahydrofuran:2-propanol:2,2,4-trimethylpentane solution; crystals of (3), m.p. 503–505 K, and (4), m.p. 485–488 K, were prepared by a modification of the method followed by Akhtar, Fray & Yarrow (1968) and were grown by slow cooling of a 1:3 2,2,4-trimethylpentane:2-propanol solution and slow evaporation of a 4:1 hexanes:ethyl acetate solution, respectively.



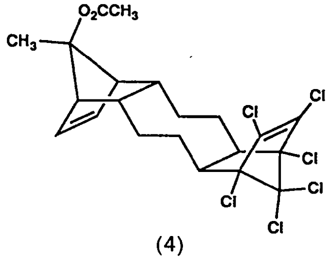
(1)



(2)



(3)



(4)

The asymmetric unit of (1) consists of four molecules: *A* and *B* in the twist-boat conformation and *C* and *D* in the chair conformation. The four molecules are arranged such that their OH groups point inwards towards a central point (at 0.22, 0.02, 0.75), forming four hydrogen bonds in the shape of a rhombus. The O···O distances are in the range 2.763 (3)–2.788 (2) Å and the O···O···O angles are 91.89 (7)° at O1*A*, 88.38 (6)° at O1*B*, 91.66 (7)° at O1*C* and 88.07 (6)° at O1*D*. The angles about H in these four hydrogen bonds range from 163 (2) to 170 (2)°. Structural data for *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 (Garcia, Fronczek & McLaughlin, 1991*a*), *endo,endo,anti*-17,17,18,18-tetramethoxypentacyclo[12.2.1.$1^{6,9}$.$0^{2,13}$.$0^{5,10}$]octadeca-7,15-diene (Garcia, Fronczek & McLaughlin, 1991*a*), *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 (Garcia, Fronczek & McLaughlin, 1991*b*), *endo*-tricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-13-one (Garcia, Fronczek & McLaughlin, 1991*c*), *endo,endo,anti*-pentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene-17,18-dione (Garcia, Fronczek & McLaughlin, 1991*d*), *endo,endo,anti*-1,14,15,16,17,17-hexachloro-18,18-diethoxypentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene (Garcia, Fronczek & McLaughlin, 1994, *endo*-1,10,11,12,13,13-hexachlorotricyclo[8.2.1.0^{2,9}]trideca-7,11-diene (Garcia, Fronczek & McLaughlin, 1994), *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 (Garcia, Fronczek & McLaughlin, 1992*a*), *endo,endo,endo,anti*-1,8,9,10,11,18,19,20,21,21,23,23-dodecachloroheptacyclo[16.2.1.1^{3,16}.1^{8,11}.0^{2,17}.0^{4,15}.0^{7,12}]tricosa-9,19-diene (Garcia, Fronczek & McLaughlin, 1992*b*) and *endo*-1,10,11,12-tetrachloro-13,13-dimethoxytricyclo[8.2.1.0^{2,9}]trideca-5,11-diene (Li, Fronczek & McLaughlin, 1992) are in agreement with those of the title compounds.

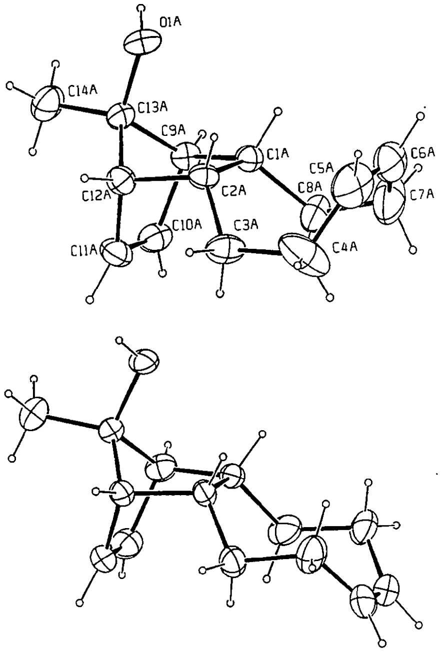


Fig. 1. *ORTEP* drawing (Johnson, 1965) of (1), representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radii; one of the two twist-boat-conformation molecules and one of the chair-conformation molecules are illustrated.

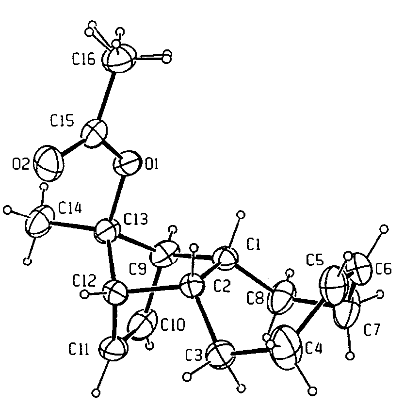


Fig. 2. *ORTEP* drawing (Johnson, 1965) of (2), representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radii.

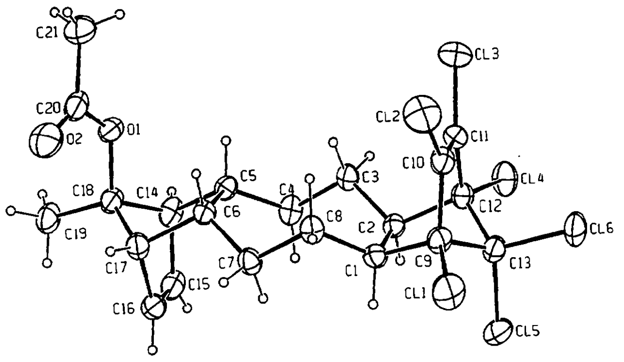


Fig. 3. *ORTEP* drawing (Johnson, 1965) of (3), representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radii.

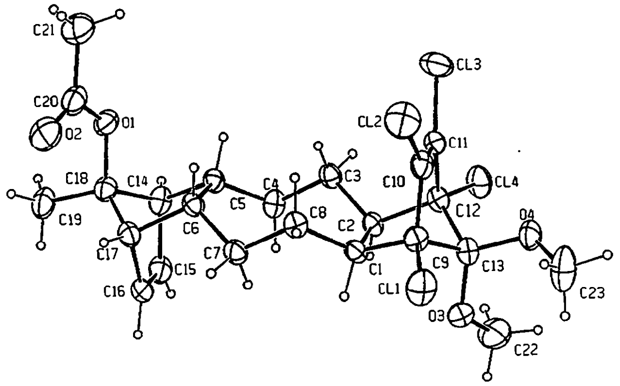


Fig. 4. *ORTEP* drawing (Johnson, 1965) of (4), representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radii.

The cyclooctene rings in (1) (two of the four independent molecules) and (2) have twist-boat conformations, with torsion angles ω_1 C8—C1—C2—C3, C4—C5—C6—C7, ω_2 C1—C2—C3—C4, C2—C1—C8—C7, ω_3 C2—C3—C4—C5, C1—C8—C7—C6 of $\omega_1 = -1.1$ (4), 1.5 (7), $\omega_2 = 63.0$ (4), −82.1 (4), $\omega_3 = 6.2$ (6), 25.7 (5)° for molecule *A* of (1), $\omega_1 = 1.6$ (4), −0.2 (5), $\omega_2 = 75.6$ (4), −68.7 (4), $\omega_3 = -14.1$ (5), −1.0 (4)° for molecule *B* of (1) and $\omega_1 = -0.1$ (3), −1.4 (4), $\omega_2 = 83.5$ (2), −60.4 (3), $\omega_3 =$

−27.8 (3), −10.0 (3)° for (2), which are distorted from the ideal boat D_{2d} conformation (of cyclooctane) with torsion angles $\omega_1 = \pm 0$, $\omega_2 = \pm 76.4$ and $\omega_3 = \pm 0°$ (Hendrickson, 1964) and $\omega_1 = \pm 0$, $\omega_2 = \pm 73.4$ and $\omega_3 = \pm 0°$ (Ivanov & Osawa, 1984).

Experimental

Compound (1)

Crystal data

$C_{14}H_{20}O$
M_r = 204.3
Triclinic
$P\bar{1}$
a = 11.575 (3) Å
b = 14.1480 (13) Å
c = 17.132 (3) Å
α = 77.97 (1)°
β = 70.28 (2)°
γ = 70.56 (1)°
V = 2475.8 (9) Å^3
Z = 8

D_x = 1.096 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 9–12°
μ = 0.06 mm^{-1}
T = 297 K
Parallelepiped
0.55 × 0.52 × 0.32 mm
Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: none
8688 measured reflections
8688 independent reflections
5262 observed reflections [$I > 2\sigma(I)$]

$\theta_{\max}$ = 25°
$h = 0 \rightarrow 13$
$k = -15 \rightarrow 16$
$l = -18 \rightarrow 20$
3 standard reflections
frequency: 166.67 min
intensity variation: −4.6% (linear correction)

Refinement

Refinement on F
R = 0.063
wR = 0.066
S = 2.839
5262 reflections
558 parameters
H atoms refined isotropically
$w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$
$(\Delta/\sigma)_{\max}$ = 0.05

$\Delta\rho_{\max}$ = 0.36 e Å^{-3}
$\Delta\rho_{\min}$ = −0.09 e Å^{-3}
Extinction correction: $(1 + gI_c)^{-1}$ applied to F_c
Extinction coefficient: 3.9 (3) × 10^{-7}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (1)

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

	x	y	z	$B_{\rm eq}$
O1*A*	0.0873 (2)	−0.0526 (1)	0.7982 (1)	5.29 (5)
C1*A*	−0.0305 (2)	−0.1142 (2)	0.7128 (2)	4.30 (7)
C2*A*	0.0579 (2)	−0.2104 (2)	0.7494 (2)	4.32 (7)
C3*A*	0.0645 (3)	−0.3143 (2)	0.7320 (2)	6.6 (1)
C4*A*	0.1160 (5)	−0.3419 (3)	0.6424 (3)	13.1 (2)
C5*A*	0.1690 (4)	−0.2700 (3)	0.5785 (2)	10.1 (1)
C6*A*	0.1027 (3)	−0.1823 (3)	0.5463 (2)	8.6 (1)
C7*A*	−0.0352 (4)	−0.1411 (4)	0.5706 (2)	11.5 (2)
C8*A*	−0.1053 (3)	−0.1277 (2)	0.6594 (2)	6.5 (1)
C9*A*	−0.1184 (2)	−0.0628 (2)	0.7919 (2)	4.27 (7)
C10*A*	−0.2003 (3)	−0.1291 (2)	0.8427 (2)	5.66 (8)
C11*A*	−0.1265 (3)	−0.2090 (2)	0.8740 (2)	6.02 (9)
C12*A*	0.0073 (3)	−0.1979 (2)	0.8435 (2)	4.93 (8)
C13*A*	−0.0248 (2)	−0.0836 (2)	0.8433 (2)	4.39 (7)
C14*A*	−0.0804 (3)	−0.0419 (2)	0.9269 (2)	7.4 (1)
O1*B*	0.0856 (2)	0.1470 (1)	0.7751 (1)	4.46 (5)
C1*B*	−0.1330 (2)	0.2926 (2)	0.8321 (2)	3.98 (7)
C2*B*	−0.0245 (2)	0.3459 (2)	0.7986 (2)	3.71 (7)
C3*B*	−0.0596 (3)	0.4580 (2)	0.8071 (2)	5.70 (9)
C4*B*	−0.0847 (3)	0.4868 (2)	0.8925 (2)	6.7 (1)
C5*B*	−0.1041 (3)	0.4101 (2)	0.9649 (2)	5.97 (9)
C6*B*	−0.1998 (3)	0.3704 (2)	0.9902 (2)	6.3 (1)
C7*B*	−0.3021 (3)	0.3956 (3)	0.9512 (2)	7.4 (1)
C8*B*	−0.2703 (3)	0.3556 (2)	0.8699 (2)	6.3 (1)
C9*B*	−0.1197 (2)	0.2452 (2)	0.7548 (2)	4.70 (7)
C10*B*	−0.1578 (3)	0.3306 (2)	0.6913 (2)	6.00 (9)
C11*B*	−0.0672 (3)	0.3761 (2)	0.6630 (2)	5.77 (9)
C12*B*	0.0333 (2)	0.3216 (2)	0.7065 (2)	4.32 (7)
C13*B*	0.0255 (2)	0.2133 (2)	0.7151 (2)	3.92 (7)
C14*B*	0.0738 (3)	0.1630 (2)	0.6356 (2)	6.15 (9)
O1*C*	0.3501 (2)	0.0849 (1)	0.7048 (1)	5.60 (5)
C1*C*	0.3755 (2)	0.2419 (2)	0.7587 (2)	4.55 (7)
C2*C*	0.4577 (2)	0.1447 (2)	0.7983 (2)	4.26 (7)
C3*C*	0.5242 (3)	0.1537 (2)	0.8585 (2)	6.06 (9)
C4*C*	0.4332 (4)	0.1806 (3)	0.9454 (2)	8.5 (1)
C5*C*	0.4156 (3)	0.2836 (3)	0.9618 (2)	8.9 (1)
C6*C*	0.3506 (3)	0.3673 (3)	0.9274 (2)	9.1 (1)
C7*C*	0.2815 (3)	0.3716 (3)	0.8677 (2)	9.0 (1)
C8*C*	0.3670 (3)	0.3447 (2)	0.7785 (2)	6.6 (1)
C9*C*	0.4330 (3)	0.2291 (2)	0.6651 (2)	5.30 (8)
C10*C*	0.5671 (3)	0.2386 (2)	0.6401 (2)	6.24 (9)
C11*C*	0.6362 (3)	0.1577 (2)	0.6738 (2)	5.58 (9)
C12*C*	0.5511 (2)	0.0925 (2)	0.7210 (2)	4.38 (7)
C13*C*	0.4644 (2)	0.1151 (2)	0.6645 (2)	4.59 (8)
C14*C*	0.5266 (3)	0.0732 (2)	0.5806 (2)	7.2 (1)
O1*D*	0.3519 (2)	−0.1156 (1)	0.7274 (1)	4.64 (5)
C1*D*	0.4655 (2)	−0.3103 (2)	0.6902 (2)	3.87 (7)
C2*D*	0.5742 (2)	−0.2567 (2)	0.6614 (2)	3.98 (7)
C3*D*	0.7114 (3)	−0.3163 (2)	0.6198 (2)	5.54 (9)
C4*D*	0.7382 (3)	−0.3352 (2)	0.5290 (2)	7.5 (1)
C5*D*	0.7549 (3)	−0.4391 (3)	0.5155 (2)	6.8 (1)
C6*D*	0.6638 (3)	−0.4827 (2)	0.5383 (2)	6.7 (1)
C7*D*	0.5272 (3)	−0.4346 (2)	0.5814 (2)	7.0 (1)
C8*D*	0.4998 (3)	−0.4196 (2)	0.6731 (2)	5.47 (8)
C9*D*	0.4086 (3)	−0.2958 (2)	0.7837 (2)	4.45 (8)
C10*D*	0.5110 (3)	−0.3550 (2)	0.8247 (2)	5.9 (1)
C11*D*	0.5994 (3)	−0.3084 (2)	0.8009 (2)	5.97 (9)
C12*D*	0.5601 (2)	−0.2176 (2)	0.7426 (2)	4.76 (8)
C13*D*	0.4143 (2)	−0.1881 (2)	0.7835 (2)	4.32 (7)
C14*D*	0.3654 (3)	−0.1465 (2)	0.8672 (2)	6.6 (1)

Table 2. *Selected geometric parameters* (Å, °) *for* (1)

O1*A*—C13*A*	1.426 (3)	O1*C*—C13*C*	1.431 (3)
O1*A*—HOH*A*	0.77 (2)	O1*C*—HOH*C*	0.74 (2)
C1*A*—C2*A*	1.564 (3)	C1*C*—C2*C*	1.560 (3)
C1*A*—C8*A*	1.530 (5)	C1*C*—C8*C*	1.526 (4)
C1*A*—C9*A*	1.546 (3)	C1*C*—C9*C*	1.539 (4)
C2*A*—C3*A*	1.532 (4)	C2*C*—C3*C*	1.526 (5)
C2*A*—C12*A*	1.544 (4)	C2*C*—C12*C*	1.542 (3)
C3*A*—C4*A*	1.529 (6)	C3*C*—C4*C*	1.543 (4)
C4*A*—C5*A*	1.452 (6)	C4*C*—C5*C*	1.476 (6)
C5*A*—C6*A*	1.342 (5)	C5*C*—C6*C*	1.315 (5)
C6*A*—C7*A*	1.447 (5)	C6*C*—C7*C*	1.477 (6)
C7*A*—C8*A*	1.481 (5)	C7*C*—C8*C*	1.558 (5)
C9*A*—C10*A*	1.493 (4)	C9*C*—C10*C*	1.509 (5)
C9*A*—C13*A*	1.536 (4)	C9*C*—C13*C*	1.531 (4)
C10*A*—C11*A*	1.307 (4)	C10*C*—C11*C*	1.308 (4)
C11*A*—C12*A*	1.509 (5)	C11*C*—C12*C*	1.494 (4)
C12*A*—C13*A*	1.533 (4)	C12*C*—C13*C*	1.536 (5)
C13*A*—C14*A*	1.515 (4)	C13*C*—C14*C*	1.519 (4)
O1*B*—C13*B*	1.438 (3)	O1*D*—C13*D*	1.436 (3)
O1*B*—HOH*B*	0.73 (2)	O1*D*—HOH*D*	0.79 (3)
C1*B*—C2*B*	1.561 (4)	C1*D*—C2*D*	1.569 (4)
C1*B*—C8*B*	1.531 (3)	C1*D*—C8*D*	1.529 (4)

C1*B*—C9*B*	1.545 (4)	C1*D*—C9*D*	1.541 (4)
C2*B*—C3*B*	1.526 (4)	C2*D*—C3*D*	1.529 (3)
C2*B*—C12*B*	1.553 (4)	C2*D*—C12*D*	1.541 (4)
C3*B*—C4*B*	1.507 (5)	C3*D*—C4*D*	1.542 (5)
C4*B*—C5*B*	1.476 (4)	C4*D*—C5*D*	1.474 (5)
C5*B*—C6*B*	1.312 (5)	C5*D*—C6*D*	1.303 (6)
C6*B*—C7*B*	1.458 (5)	C6*D*—C7*D*	1.488 (4)
C7*B*—C8*B*	1.497 (5)	C7*D*—C8*D*	1.540 (5)
C9*B*—C10*B*	1.503 (4)	C9*D*—C10*D*	1.502 (4)
C9*B*—C13*B*	1.531 (3)	C9*D*—C13*D*	1.546 (4)
C10*B*—C11*B*	1.313 (5)	C10*D*—C11*D*	1.305 (5)
C11*B*—C12*B*	1.501 (4)	C11*D*—C12*D*	1.497 (4)
C12*B*—C13*B*	1.538 (4)	C12*D*—C13*D*	1.539 (3)
C13*B*—C14*B*	1.515 (4)	C13*D*—C14*D*	1.518 (4)

C13*A*—O1*A*—HOH*A*	111 (2)	C13*C*—O1*C*—HOH*C*	111 (2)
C2*A*—C1*A*—C8*A*	118.4 (2)	C2*C*—C1*C*—C8*C*	119.3 (3)
C2*A*—C1*A*—C9*A*	102.2 (2)	C2*C*—C1*C*—C9*C*	101.9 (2)
C8*A*—C1*A*—C9*A*	112.8 (2)	C8*C*—C1*C*—C9*C*	112.9 (2)
C1*A*—C2*A*—C3*A*	119.0 (3)	C1*C*—C2*C*—C3*C*	119.6 (3)
C1*A*—C2*A*—C12*A*	101.8 (2)	C1*C*—C2*C*—C12*C*	102.3 (2)
C3*A*—C2*A*—C12*A*	112.4 (2)	C3*C*—C2*C*—C12*C*	113.4 (2)
C2*A*—C3*A*—C4*A*	120.0 (3)	C2*C*—C3*C*—C4*C*	114.3 (3)
C3*A*—C4*A*—C5*A*	116.7 (4)	C3*C*—C4*C*—C5*C*	113.9 (3)
C4*A*—C5*A*—C6*A*	126.5 (4)	C4*C*—C5*C*—C6*C*	125.5 (4)
C5*A*—C6*A*—C7*A*	126.0 (4)	C5*C*—C6*C*—C7*C*	124.6 (4)
C6*A*—C7*A*—C8*A*	119.3 (4)	C6*C*—C7*C*—C8*C*	115.8 (3)
C1*A*—C8*A*—C7*A*	116.7 (3)	C1*C*—C8*C*—C7*C*	115.0 (3)
C1*A*—C9*A*—C10*A*	107.2 (2)	C1*C*—C9*C*—C10*C*	106.7 (3)
C1*A*—C9*A*—C13*A*	101.7 (2)	C1*C*—C9*C*—C13*C*	102.5 (2)
C10*A*—C9*A*—C13*A*	100.5 (2)	C10*C*—C9*C*—C13*C*	99.4 (2)
C9*A*—C10*A*—C11*A*	107.2 (3)	C9*C*—C10*C*—C11*C*	107.5 (3)
C10*A*—C11*A*—C12*A*	107.6 (3)	C10*C*—C11*C*—C12*C*	107.4 (3)
C2*A*—C12*A*—C11*A*	107.0 (3)	C2*C*—C12*C*—C11*C*	107.1 (2)
C2*A*—C12*A*—C13*A*	102.3 (2)	C2*C*—C12*C*—C13*C*	101.7 (2)
C11*A*—C12*A*—C13*A*	99.5 (2)	C11*C*—C12*C*—C13*C*	100.1 (2)
O1*A*—C13*A*—C9*A*	113.1 (2)	O1*C*—C13*C*—C9*C*	109.1 (2)
O1*A*—C13*A*—C12*A*	108.9 (2)	O1*C*—C13*C*—C12*C*	112.9 (2)
O1*A*—C13*A*—C14*A*	108.7 (3)	O1*C*—C13*C*—C14*C*	108.2 (3)
C9*A*—C13*A*—C12*A*	91.9 (2)	C9*C*—C13*C*—C12*C*	92.1 (2)
C9*A*—C13*A*—C14*A*	115.9 (2)	C9*C*—C13*C*—C14*C*	117.6 (2)
C12*A*—C13*A*—C14*A*	117.5 (2)	C12*C*—C13*C*—C14*C*	116.2 (2)
C13*B*—O1*B*—HOH*B*	109 (1)	C13*D*—O1*D*—HOH*D*	110 (2)
C2*B*—C1*B*—C8*B*	118.6 (2)	C2*D*—C1*D*—C8*D*	118.8 (2)
C2*B*—C1*B*—C9*B*	102.3 (2)	C2*D*—C1*D*—C9*D*	102.5 (2)
C8*B*—C1*B*—C9*B*	112.2 (3)	C8*D*—C1*D*—C9*D*	113.3 (2)
C1*B*—C2*B*—C3*B*	118.5 (2)	C1*D*—C2*D*—C3*D*	119.1 (2)
C1*B*—C2*B*—C12*B*	102.0 (2)	C1*D*—C2*D*—C12*D*	101.9 (2)
C3*B*—C2*B*—C12*B*	113.1 (2)	C3*D*—C2*D*—C12*D*	113.0 (3)
C2*B*—C3*B*—C4*B*	117.2 (2)	C2*D*—C3*D*—C4*D*	115.2 (3)
C3*B*—C4*B*—C5*B*	118.3 (3)	C3*D*—C4*D*—C5*D*	116.3 (3)
C4*B*—C5*B*—C6*B*	124.5 (3)	C4*D*—C5*D*—C6*D*	125.2 (3)
C5*B*—C6*B*—C7*B*	125.0 (3)	C5*D*—C6*D*—C7*D*	125.0 (3)
C6*B*—C7*B*—C8*B*	117.5 (2)	C6*D*—C7*D*—C8*D*	114.8 (3)
C1*B*—C8*B*—C7*B*	118.4 (3)	C1*D*—C8*D*—C7*D*	115.2 (2)
C1*B*—C9*B*—C10*B*	107.0 (2)	C1*D*—C9*D*—C10*D*	106.9 (2)
C1*B*—C9*B*—C13*B*	102.2 (2)	C1*D*—C9*D*—C13*D*	101.6 (2)
C10*B*—C9*B*—C13*B*	99.7 (2)	C10*D*—C9*D*—C13*D*	99.4 (3)
C9*B*—C10*B*—C11*B*	107.4 (3)	C9*D*—C10*D*—C11*D*	107.7 (2)
C10*B*—C11*B*—C12*B*	107.5 (2)	C10*D*—C11*D*—C12*D*	107.8 (3)
C2*B*—C12*B*—C11*B*	107.1 (2)	C2*D*—C12*D*—C11*D*	106.6 (2)
C2*B*—C12*B*—C13*B*	101.6 (2)	C2*D*—C12*D*—C13*D*	102.6 (3)
C11*B*—C12*B*—C13*B*	99.7 (3)	C11*D*—C12*D*—C13*D*	99.5 (2)
O1*B*—C13*B*—C9*B*	109.2 (2)	O1*D*—C13*D*—C9*D*	112.5 (3)
O1*B*—C13*B*—C12*B*	112.5 (3)	O1*D*—C13*D*—C12*D*	109.6 (2)
O1*B*—C13*B*—C14*B*	108.6 (2)	O1*D*—C13*D*—C14*D*	108.1 (2)
C9*B*—C13*B*—C12*B*	92.3 (2)	C9*D*—C13*D*—C12*D*	92.1 (2)
C9*B*—C13*B*—C14*B*	116.4 (3)	C9*D*—C13*D*—C14*D*	117.1 (2)
C12*B*—C13*B*—C14*B*	117.0 (2)	C12*D*—C13*D*—C14*D*	116.8 (3)

C8*A*—C1*A*—C2*A*—C3*A*	−1.1 (4)	C8*C*—C1*C*—C2*C*—C3*C*	2.2 (4)
C6*A*—C7*A*—C8*A*—C1*A*	25.7 (5)	C6*C*—C7*C*—C8*C*—C1*C*	106.3 (4)
C4*A*—C5*A*—C6*A*—C7*A*	1.5 (7)	C4*C*—C5*C*—C6*C*—C7*C*	1.5 (6)
C2*A*—C3*A*—C4*A*—C5*A*	6.2 (6)	C2*C*—C3*C*—C4*C*—C5*C*	−109.6 (4)
C8*B*—C1*B*—C2*B*—C3*B*	1.6 (4)	C8*D*—C1*D*—C2*D*—C3*D*	−1.9 (4)
C6*B*—C7*B*—C8*B*—C1*B*	−1.0 (4)	C6*D*—C7*D*—C8*D*—C1*D*	109.0 (3)
C4*B*—C5*B*—C6*B*—C7*B*	−0.2 (5)	C4*D*—C5*D*—C6*D*—C7*D*	0.3 (5)
C2*B*—C3*B*—C4*B*—C5*B*	−14.1 (5)	C2*D*—C3*D*—C4*D*—C5*D*	−107.3 (4)

Compound (2)

Crystal data

$C_{16}H_{22}O_2$
$M_r = 246.4$
Monoclinic
$P2_1/n$
$a = 8.8764\ (4)$ Å
$b = 18.1005\ (14)$ Å
$c = 8.9616\ (5)$ Å
$\beta = 107.514\ (4)°$
$V = 1373.1\ (3)$ Å^3
$Z = 4$

$D_x = 1.192$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10-13°$
$\mu = 0.07$ mm^{-1}
$T = 295$ K
Prism
0.38 × 0.32 × 0.25 mm
Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: none
3782 measured reflections
3462 independent reflections
2293 observed reflections [$I > \sigma(I)$]

$R_{\rm int} = 0.016$
$\theta_{\rm max} = 28.5°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 24$
$l = -12 \rightarrow 11$
3 standard reflections
frequency: 166.67 min
intensity variation: <2%

Refinement

Refinement on F
$R = 0.064$
$wR = 0.055$
$S = 2.299$
2293 reflections
224 parameters
H atoms refined isotropically
$w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$
$(\Delta/\sigma)_{\rm max} = 0.01$

$\Delta\rho_{\rm max} = 0.23$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.09$ e Å^{-3}
Extinction correction: $(1 + gI_c)^{-1}$ applied to F_c
Extinction coefficient: $6.2\ (13) \times 10^{-7}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (2)

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

	x	y	z	$B_{\rm eq}$
O1	0.6711 (1)	0.49030 (7)	0.7727 (1)	3.64 (3)
O2	0.8122 (2)	0.48113 (9)	1.0264 (1)	5.17 (4)
C1	0.6948 (2)	0.3987 (1)	0.5422 (2)	3.28 (4)
C2	0.8222 (2)	0.3686 (1)	0.6918 (2)	3.19 (4)
C3	0.9201 (2)	0.3025 (1)	0.6741 (2)	4.75 (5)
C4	1.0625 (2)	0.3192 (2)	0.6207 (3)	7.26 (7)
C5	1.0603 (2)	0.3850 (1)	0.5237 (2)	5.45 (6)
C6	0.9521 (2)	0.4027 (1)	0.3955 (2)	5.07 (5)
C7	0.8101 (2)	0.3586 (2)	0.3197 (2)	6.46 (6)
C8	0.6734 (2)	0.3612 (1)	0.3848 (2)	5.12 (5)
C9	0.5402 (2)	0.3959 (1)	0.5917 (2)	3.97 (4)
C10	0.5052 (2)	0.3159 (1)	0.6087 (2)	5.25 (5)
C11	0.6118 (2)	0.2905 (1)	0.7335 (2)	5.14 (5)
C12	0.7219 (2)	0.3528 (1)	0.8040 (2)	3.62 (4)
C13	0.6019 (2)	0.4165 (1)	0.7665 (2)	3.59 (4)
C14	0.4815 (2)	0.4193 (1)	0.8556 (2)	5.15 (5)
C15	0.7679 (2)	0.5165 (1)	0.9080 (2)	3.62 (4)
C16	0.8118 (2)	0.5944 (1)	0.8908 (2)	5.07 (5)

Table 4. *Selected geometric parameters* (Å, °) *for* (2)

O1—C13	1.464 (2)	C5—C6	1.296 (2)
O1—C15	1.344 (2)	C6—C7	1.474 (3)
O2—C15	1.199 (2)	C7—C8	1.497 (3)

C1—C2	1.569 (2)	C9—C10	1.500 (3)
C1—C8	1.525 (3)	C10—C11	1.312 (2)
C2—C3	1.516 (3)	C11—C12	1.502 (3)
C2—C12	1.556 (3)	C12—C13	1.536 (3)
C3—C4	1.511 (3)	C13—C14	1.516 (3)
C4—C5	1.472 (4)	C15—C16	1.483 (3)
C13—O1—C15	120.2 (1)	C10—C9—C13	98.7 (1)
C2—C1—C8	119.5 (2)	C9—C10—C11	107.8 (2)
C2—C1—C9	102.4 (1)	C10—C11—C12	107.8 (2)
C8—C1—C9	111.8 (1)	C2—C12—C11	107.0 (1)
C1—C2—C3	118.2 (1)	C2—C12—C13	102.4 (1)
C1—C2—C12	102.2 (1)	C11—C12—C13	98.7 (1)
C3—C2—C12	112.3 (2)	O1—C13—C9	106.0 (1)
C2—C3—C4	115.9 (2)	O1—C13—C12	114.9 (1)
C3—C4—C5	119.0 (2)	O1—C13—C14	107.7 (2)
C4—C5—C6	126.7 (2)	C9—C13—C12	93.0 (1)
C5—C6—C7	125.3 (2)	C9—C13—C14	116.9 (1)
C6—C7—C8	119.0 (2)	C12—C13—C14	117.5 (2)
C1—C8—C7	119.0 (2)	O1—C15—O2	124.1 (2)
C1—C9—C10	106.7 (2)	O1—C15—C16	111.0 (1)
C1—C9—C13	101.9 (1)	O2—C15—C16	125.0 (2)
C8—C1—C2—C3	−0.1 (3)	C6—C7—C8—C1	−10.0 (3)
C4—C5—C6—C7	−1.4 (4)	C2—C3—C4—C5	−27.8 (3)

Compound (3)

Crystal data

$C_{23}H_{28}Cl_4O_4$
$M_r = 510.3$
Monoclinic
$P2_1/c$
$a = 12.9664\ (9)$ Å
$b = 11.3814\ (10)$ Å
$c = 16.6962\ (14)$ Å
$\beta = 105.549\ (6)°$
$V = 2373.8\ (7)$ Å^3
$Z = 4$

$D_x = 1.428$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10\text{-}12°$
$\mu = 0.53$ mm^{-1}
$T = 296$ K
Prism
0.38 × 0.15 × 0.15 mm
Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: empirical
$T_{\min} = 0.975$, $T_{\max} = 1.000$
4582 measured reflections
4158 independent reflections
2541 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.017$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 13$
$l = -19 \rightarrow 19$
3 standard reflections
frequency: 166.67 min
intensity variation: <2%

Refinement

Refinement on F
$R = 0.039$
$wR = 0.039$
$S = 1.580$
4158 reflections
281 parameters
H-atom parameters not refined
$w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$
$(\Delta/\sigma)_{\max} = 0.01$

$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.10$ e Å^{-3}
Extinction correction: $(1 + gI_c)^{-1}$ applied to F_c
Extinction coefficient: $4\ (2) \times 10^{-8}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 5. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (3)

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

	x	y	z	$B_{\rm eq}$
Cl1	0.71217 (8)	0.90651 (9)	0.63144 (5)	4.06 (2)
Cl2	0.48785 (6)	0.7534 (1)	0.55502 (6)	4.61 (2)
Cl3	0.51285 (7)	0.5965 (1)	0.38880 (6)	4.51 (2)
Cl4	0.74357 (7)	0.65526 (9)	0.35303 (5)	3.64 (2)
O1	0.7232 (2)	0.1690 (2)	0.7268 (1)	3.11 (5)
O2	0.6814 (2)	0.2125 (3)	0.8454 (1)	4.61 (6)
O3	0.8615 (2)	0.8400 (2)	0.5175 (1)	3.11 (5)
O4	0.7006 (2)	0.8931 (2)	0.4237 (1)	3.29 (5)
C1	0.7760 (2)	0.6821 (3)	0.6018 (2)	2.33 (7)
C2	0.7893 (2)	0.6105 (3)	0.5243 (2)	2.18 (7)
C3	0.7567 (2)	0.4813 (3)	0.5180 (2)	2.43 (7)
C4	0.8385 (2)	0.3993 (3)	0.5744 (2)	2.77 (7)
C5	0.7981 (2)	0.3318 (3)	0.6392 (2)	2.28 (7)
C6	0.7806 (2)	0.4030 (3)	0.7154 (2)	2.29 (7)
C7	0.8074 (2)	0.5344 (3)	0.7229 (2)	2.66 (7)
C8	0.7288 (2)	0.6170 (3)	0.6639 (2)	2.40 (7)
C9	0.7073 (2)	0.7890 (3)	0.5613 (2)	2.49 (7)
C10	0.5971 (2)	0.7440 (3)	0.5174 (2)	2.71 (7)
C11	0.6077 (2)	0.6827 (3)	0.4528 (2)	2.58 (7)
C12	0.7242 (2)	0.6872 (3)	0.4516 (2)	2.37 (7)
C13	0.7528 (2)	0.8137 (3)	0.4851 (2)	2.74 (7)
C14	0.8759 (2)	0.2329 (3)	0.6826 (2)	2.81 (7)
C15	0.9806 (2)	0.2912 (3)	0.7248 (2)	3.32 (8)
C16	0.9658 (2)	0.3500 (3)	0.7888 (2)	3.27 (8)
C17	0.8511 (2)	0.3326 (3)	0.7907 (2)	2.60 (7)
C18	0.8347 (2)	0.2054 (3)	0.7589 (2)	2.68 (7)
C19	0.8926 (3)	0.1098 (3)	0.8177 (2)	3.78 (9)
C20	0.6570 (3)	0.1737 (3)	0.7758 (2)	3.48 (8)
C21	0.5500 (3)	0.1247 (4)	0.7321 (3)	5.4 (13
C22	0.9247 (3)	0.8492 (4)	0.4606 (2)	5.0 (1)
C23	0.6968 (4)	1.0109 (4)	0.4459 (3)	5.9 (1)

Table 6. *Selected geometric parameters* (Å, °) *for* (3)

Cl1—C9	1.768 (3)	C5—C6	1.574 (4)
Cl2—C10	1.699 (3)	C5—C14	1.555 (4)
Cl3—C11	1.707 (3)	C6—C7	1.533 (5)
Cl4—C12	1.768 (3)	C6—C17	1.563 (4)
O1—C18	1.461 (3)	C7—C8	1.534 (4)
O1—C20	1.335 (4)	C9—C10	1.510 (4)
O2—C20	1.203 (4)	C9—C13	1.565 (5)
O3—C13	1.401 (3)	C10—C11	1.322 (5)
O3—C22	1.415 (5)	C11—C12	1.516 (4)
O4—C13	1.397 (4)	C12—C13	1.553 (5)
O4—C23	1.397 (5)	C14—C15	1.506 (4)
C1—C2	1.578 (4)	C14—C18	1.540 (5)
C1—C8	1.528 (5)	C15—C16	1.317 (5)
C1—C9	1.552 (4)	C16—C17	1.510 (4)
C2—C3	1.527 (5)	C17—C18	1.537 (5)
C2—C12	1.549 (4)	C18—C19	1.522 (5)
C3—C4	1.532 (4)	C20—C21	1.493 (5)
C4—C5	1.529 (5)		
C18—O1—C20	120.0 (2)	C1—C9—C10	107.6 (3)
C13—O3—C22	117.4 (2)	C1—C9—C13	101.5 (2)
C13—O4—C23	118.1 (2)	C10—C9—C13	100.4 (2)
C2—C1—C8	117.2 (3)	Cl2—C10—C9	125.0 (2)
C2—C1—C9	102.9 (2)	Cl2—C10—C11	127.9 (2)
C8—C1—C9	112.9 (3)	C9—C10—C11	106.4 (3)
C1—C2—C3	117.7 (3)	Cl3—C11—C10	126.8 (3)
C1—C2—C12	101.3 (2)	Cl3—C11—C12	124.3 (2)
C3—C2—C12	114.0 (2)	C10—C11—C12	108.4 (2)
C2—C3—C4	114.3 (2)	Cl4—C12—C2	115.5 (2)
C3—C4—C5	115.4 (3)	Cl4—C12—C11	113.3 (2)
C4—C5—C6	117.6 (3)	Cl4—C12—C13	116.5 (2)
C4—C5—C14	113.0 (3)	C2—C12—C11	107.8 (3)
C6—C5—C14	102.3 (2)	C2—C12—C13	102.3 (2)
C5—C6—C7	119.3 (3)	C11—C12—C13	99.7 (3)
C5—C6—C17	101.9 (2)	O3—C13—O4	112.6 (3)
C7—C6—C17	111.3 (2)	O3—C13—C9	106.5 (2)
C6—C7—C8	116.6 (2)	O3—C13—C12	117.1 (3)
C1—C8—C7	114.5 (3)	O4—C13—C9	119.9 (3)

Cl1—C9—C1	113.0 (2)	O4—C13—C12	108.3 (2)
Cl1—C9—C10	116.0 (2)	C9—C13—C12	91.2 (2)
Cl1—C9—C13	116.7 (2)	C5—C14—C15	106.8 (3)
C5—C14—C18	101.9 (3)	O1—C18—C17	115.1 (3)
C15—C14—C18	99.8 (2)	O1—C18—C19	107.6 (3)
C14—C15—C16	107.2 (3)	C14—C18—C17	92.8 (3)
C15—C16—C17	108.0 (3)	C14—C18—C19	117.3 (3)
C6—C17—C16	106.4 (3)	C17—C18—C19	116.9 (2)
C6—C17—C18	102.0 (2)	O1—C20—O2	124.1 (3)
C16—C17—C18	99.3 (3)	O1—C20—C21	110.6 (3)
O1—C18—C14	106.5 (2)	O2—C20—C21	125.3 (3)
C4—C5—C6—C7	−1.7 (4)	C2—C3—C4—C5	119.1 (3)
C8—C1—C2—C3	−1.4 (4)	C6—C7—C8—C1	−115.9 (3)

Compound (4)

Crystal data

$C_{21}H_{22}Cl_6O_2$
M_r = 519.1
Monoclinic
$P2_1/c$
a = 13.0246 (6) Å
b = 10.7445 (6) Å
c = 16.8103 (9) Å
β = 104.017 (4)°
V = 2282.5 (5) Å^3
Z = 4

D_x = 1.511 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 11-14°
μ = 0.77 mm^{-1}
T = 295 K
Prism with bipyramid
0.60 × 0.35 × 0.25 mm
Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: empirical
$T_{\min}$ = 0.926, $T_{\max}$ = 0.998
7228 measured reflections
6635 independent reflections
4178 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int}$ = 0.012
$\theta_{\max}$ = 30°
$h = 0 \rightarrow 18$
$k = 0 \rightarrow 15$
$l = -23 \rightarrow 22$
3 standard reflections
frequency: 166.67 min
intensity variation: <2%

Refinement

Refinement on F
R = 0.033
wR = 0.033
S = 1.519
4178 reflections
351 parameters
All H-atom parameters refined
$w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$
$(\Delta/\sigma)_{\max}$ = 0.02

$\Delta\rho_{\max}$ = 0.30 e Å^{-3}
$\Delta\rho_{\min}$ = −0.10 e Å^{-3}
Extinction correction: $(1 + gI_c)^{-1}$ applied to F_c
Extinction coefficient: 1.2 (2) × 10^{-7}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 7. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (4)

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

	x	y	z	$B_{\rm eq}$
Cl1	0.71272 (5)	0.95256 (5)	0.61124 (3)	4.09 (1)
Cl2	0.49011 (4)	0.78818 (6)	0.55355 (4)	4.46 (1)
Cl3	0.50820 (4)	0.60090 (6)	0.39524 (3)	4.03 (1)
Cl4	0.73535 (4)	0.65776 (5)	0.34672 (3)	3.64 (1)
Cl5	0.88098 (4)	0.87180 (5)	0.48637 (4)	4.12 (1)
Cl6	0.67382 (4)	0.95016 (5)	0.40280 (3)	3.77 (1)
O1	0.7297 (1)	0.1801 (1)	0.73989 (8)	2.78 (3)
O2	0.6875 (1)	0.2349 (2)	0.85680 (9)	4.62 (4)
C1	0.7757 (1)	0.7084 (2)	0.5921 (1)	2.27 (3)
C2	0.7850 (1)	0.6252 (2)	0.5169 (1)	2.16 (3)
C3	0.7540 (1)	0.4887 (2)	0.5180 (1)	2.45 (4)
C4	0.8377 (2)	0.4073 (2)	0.5751 (1)	2.71 (4)
C5	0.8005 (1)	0.3432 (2)	0.6444 (1)	2.30 (3)
C6	0.7837 (1)	0.4265 (2)	0.7173 (1)	2.12 (3)
C7	0.8109 (2)	0.5644 (2)	0.7177 (1)	2.46 (4)
C8	0.7315 (1)	0.6476 (2)	0.6588 (1)	2.44 (4)
C9	0.7063 (2)	0.8196 (2)	0.5498 (1)	2.53 (4)
C10	0.5960 (1)	0.7678 (2)	0.5131 (1)	2.54 (4)
C11	0.6034 (1)	0.6959 (2)	0.4512 (1)	2.46 (4)
C12	0.7167 (1)	0.6994 (2)	0.4436 (1)	2.29 (3)
C13	0.7443 (1)	0.8360 (2)	0.4701 (1)	2.68 (4)
C14	0.8795 (1)	0.2422 (2)	0.6895 (1)	2.80 (4)
C15	0.9820 (2)	0.3079 (2)	0.7268 (1)	3.42 (4)
C16	0.9671 (2)	0.3763 (2)	0.7877 (1)	3.39 (4)
C17	0.8541 (2)	0.3589 (2)	0.7933 (1)	2.56 (4)
C18	0.8396 (1)	0.2209 (2)	0.7678 (1)	2.55 (4)
C19	0.8976 (2)	0.1256 (2)	0.8294 (1)	3.52 (4)
C20	0.6639 (2)	0.1901 (2)	0.7898 (1)	3.29 (4)
C21	0.5577 (2)	0.1367 (3)	0.7491 (2)	5.11 (6)

Table 8. *Selected geometric parameters* (Å, °) *for* (4)

Cl1—C9	1.753 (2)	C5—C14	1.560 (2)
Cl2—C10	1.694 (2)	C6—C7	1.523 (3)
Cl3—C11	1.703 (2)	C6—C17	1.559 (2)
Cl4—C12	1.762 (2)	C7—C8	1.535 (2)
Cl5—C13	1.777 (2)	C9—C10	1.524 (2)
Cl6—C13	1.767 (2)	C9—C13	1.546 (3)
O1—C18	1.462 (2)	C10—C11	1.319 (3)
O1—C20	1.341 (3)	C11—C12	1.512 (3)
O2—C20	1.195 (2)	C12—C13	1.551 (3)
C1—C2	1.577 (3)	C14—C15	1.506 (3)
C1—C8	1.524 (3)	C14—C18	1.543 (3)
C1—C9	1.561 (2)	C15—C16	1.312 (3)
C2—C3	1.523 (3)	C16—C17	1.509 (3)
C2—C12	1.554 (2)	C17—C18	1.542 (3)
C3—C4	1.539 (2)	C18—C19	1.521 (3)
C4—C5	1.529 (3)	C20—C21	1.500 (3)
C5—C6	1.576 (3)		
C18—O1—C20	120.0 (1)	C10—C9—C13	99.1 (1)
C2—C1—C8	117.6 (1)	Cl2—C10—C9	124.8 (1)
C2—C1—C9	102.5 (1)	Cl2—C10—C11	127.9 (1)
C8—C1—C9	112.5 (2)	C9—C10—C11	107.0 (2)
C1—C2—C3	117.6 (2)	Cl3—C11—C10	127.2 (2)
C1—C2—C12	101.8 (1)	Cl3—C11—C12	124.3 (1)
C3—C2—C12	113.6 (1)	C10—C11—C12	108.1 (1)
C2—C3—C4	113.9 (1)	Cl4—C12—C2	115.0 (1)
C3—C4—C5	115.0 (2)	Cl4—C12—C11	115.3 (1)
C4—C5—C6	117.8 (1)	Cl4—C12—C13	115.6 (1)
C4—C5—C14	113.0 (2)	C2—C12—C11	107.7 (1)
C6—C5—C14	102.4 (1)	C2—C12—C13	102.1 (1)
C5—C6—C7	118.6 (2)	C11—C12—C13	99.3 (1)
C5—C6—C17	102.0 (1)	Cl5—C13—Cl6	107.1 (1)
C7—C6—C17	111.1 (1)	Cl5—C13—C9	114.2 (1)
C6—C7—C8	116.4 (1)	Cl5—C13—C12	113.7 (1)
C1—C8—C7	114.2 (2)	Cl6—C13—C9	114.1 (1)
Cl1—C9—C1	114.5 (1)	Cl6—C13—C12	115.1 (1)
Cl1—C9—C10	116.2 (1)	C9—C13—C12	92.3 (1)
Cl1—C9—C13	116.0 (1)	C5—C14—C15	106.9 (2)
C1—C9—C10	106.8 (1)	C5—C14—C18	101.5 (2)
C1—C9—C13	102.2 (2)	C15—C14—C18	99.4 (1)
C14—C15—C16	107.6 (2)	O1—C18—C19	107.6 (2)
C15—C16—C17	108.1 (2)	C14—C18—C17	92.9 (1)
C6—C17—C16	106.3 (2)	C14—C18—C19	117.8 (2)
C6—C17—C18	102.2 (1)	C17—C18—C19	116.8 (1)
C16—C17—C18	99.0 (2)	O1—C20—O2	124.3 (2)
O1—C18—C14	106.1 (1)	O1—C20—C21	110.1 (2)
O1—C18—C17	115.0 (1)	O2—C20—C21	125.6 (2)
C4—C5—C6—C7	−3.3 (2)	C2—C3—C4—C5	119.4 (2)
C8—C1—C2—C3	−2.7 (2)	C6—C7—C8—C1	−116.2 (2)

(1): The crystal was mounted in a capillary for data collection. The space group was determined by successful refinement of a centrosymmetric model. Hydroxy H atoms were located from difference maps and refined isotropically, while other H atoms were placed in calculated positions with a C—H distance of 0.95 Å and $B_{\rm iso}$ = 1.3 $B_{\rm eq}$ for the bonded C atom. The structure was solved using *SHELXS*86 (Sheldrick, 1985) and refined using *MolEN* (Fair, 1990).

(2): The crystal was mounted in a capillary for data collection. The structure was solved using *MULTAN*80 (Main *et al.*, 1980). H atoms were refined isotropically, except for those attached to C4, C7 and C16. Those on C4 and C7 did not refine well and were included as fixed contributions with a C—H distance of 0.95 Å. The methyl group involving C16 is disordered and was modeled as two half-populated rotamers with six half-populated H atoms in fixed positions.

(3): The compound is isomorphous with the hexachloro analog (4). Heavy-atom positions from (4), with the exception of Cl5 and Cl6, were successfully used as a beginning phasing model.

(4): The crystal was mounted in a capillary for data collection. The structure was solved using *MULTAN*80 (Main *et al.*, 1980).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, torsion angles for (1)-(4) and C—H bonds for (2) and (4) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71468 (155 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1071]

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2,2,4,4,6,6-Tris(2,3-naphthylenedioxy)cyclotriphosphazene

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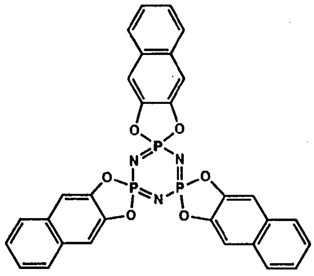
Abstract

The molecular structure of the title compound, $C_{30}H_{18}N_3O_6P_3$, is distorted from that observed in related clathrate compounds. The central phosphazene core takes a boat conformation. The peripheral naphthalene rings are not planar and the five-membered exocyclic rings are bent appreciably at the O-atom positions. The packing of the distorted molecules is governed predominantly by van der Waals forces in the crystal.

Comment

Tris(2,3-naphthylenedioxy)cyclotriphosphazene (TNDOCTP), (I), functions as a host in clathration



(I)

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