compound was crystallized by slow evaporation from ethanol (yield 70%, m.p. 423 K).

Crystal data

$C_{26}H_{36}N_4O_2$
$M_r = 436.59$
Monoclinic
$P2_1/c$
$a = 19.033\ (2)$ Å
$b = 13.661\ (1)$ Å
$c = 9.546\ (1)$ Å
$\beta = 95.72\ (1)^\circ$
$V = 2469.7\ (4)$ Å^3
$Z = 4$
$D_x = 1.174$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 40 reflections
$\theta = 5.39$–12.45°
$\mu = 0.075$ mm^{-1}
$T = 293\ (2)$ K
Prism
$0.88 \times 0.58 \times 0.36$ mm
Yellow

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
7183 measured reflections
5655 independent reflections
3065 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$

$\theta_{\max} = 27.50^\circ$
$h = -24 \rightarrow 24$
$k = -17 \rightarrow 1$
$l = -1 \rightarrow 12$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.136$
$S = 0.908$
5655 reflections
423 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0728P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.173$ e Å^{-3}
$\Delta\rho_{\min} = -0.157$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0037 (7)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

C1—O21	1.361 (2)	C20—O27	1.360 (2)
C2—C23	1.460 (2)	C23—N24	1.266 (2)
N8—C13	1.452 (2)	N24—C25	1.459 (2)
N8—C9	1.459 (2)	C29—N30	1.266 (3)
C9—C10	1.514 (2)	N30—C31	1.455 (3)
C12—C13	1.518 (2)		
O21—C1—C6	119.14 (13)	O27—C20—C15	118.9 (2)
O21—C1—C2	120.72 (13)	O27—C20—C19	121.19 (15)
C13—N8—C7	111.30 (14)	N24—C23—C2	122.9 (2)
C9—N8—C7	111.27 (13)	C23—N24—C25	118.5 (2)
C10—N11—C14	111.05 (15)	N30—C29—C19	123.2 (2)
C12—N11—C14	111.17 (14)	C29—N30—C31	117.6 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O21—H21···N24	0.97 (2)	1.71 (2)	2.604 (1)	151 (2)
O27—H27···N30	0.97 (2)	1.70 (2)	2.616 (1)	156 (2)

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1317). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). **C54**, 645–647

endo-endo-anti Diels–Alder Diadduct of Hexachlorocyclopentadiene with 1,6-Dioxacyclodeca-3,8-diene

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Abstract

There are two independent molecules of *endo-endo-anti*-1,7,8,9,10,16,17,18,19,19,20,20-dodecachloro-4,13-dioxapentacyclo[14.2.1.1^{7,10}.0^{2,15}.0^{6,11}]icosa-8,17-diene, $C_{18}H_{12}Cl_{12}O_2$, in the unit cell with different conformations. In one, the ten-membered ring adopts a chair–chair conformation, and in the other, it adopts a distorted chair–chair conformation. There are near-zero torsion angles at the ring-fusion bonds in both forms [1.5 (6) and 8.1 (5)° in the chair–chair molecule, and 2.9 (5) and 2.5 (5)° in the distorted chair–chair molecule]. The torsion angles about the bonds comprising the sides of the chair–chair are 158.5 (3), −160.7 (3), −146.8 (4) and 155.7 (3)°, and those of the distorted chair–chair are 148.0 (3), −146.8 (3), 175.7 (3) and −52.3 (4)°. The norbornene C═C bonds have lengths

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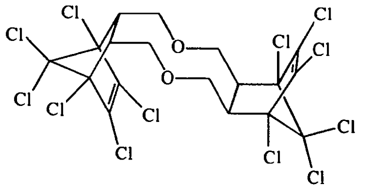
of 1.314 (6) and 1.305 (6) Å in the chair–chair molecule, and 1.309 (6) and 1.318 (6) Å in the other. The four dioxolane-system O—C bonds have average lengths of 1.411 (5) and 1.421 (5) Å in the two independent molecules.

Comment

The crystal structures of a number of Diels–Alder substituted cycloocta(e)nes and cyclohexa(e)nes have been determined in our laboratory over the past few years (Garcia & McLaughlin, 1991; Garcia, Fronczek & McLaughlin, 1991*a*,*b*,*c*, 1992*a*,*b*; Garcia, McLaughlin & Fronczek, 1991*a*,*b*; Li *et al.*, 1992; Garcia *et al.*, 1994; Garcia & Fronczek, 1995*a*,*b*). The crystal structure determination of the title compound, (I), is part of an ongoing program of structure analysis of some new derivatives bearing the dioxacyclodecane moiety, related to a study of conformation-dependent π–σ–π electronic interactions (Garcia, Fronczek & McLaughlin, 1991*a*; Garcia & McLaughlin, 1991; Garcia & Fronczek, 1995*a*,*b*).



(I)

Structural data for the title compound are in agreement with those of related compounds (Garcia, McLaughlin & Fronczek, 1991*a*; Garcia, Fronczek & McLaughlin, 1992*b*; Li *et al.*, 1992; Garcia & Fronczek, 1995*a*,*b*; Ermer *et al.*, 1973).

The dioxacyclodecane rings have chair–chair (molecule *A*) and distorted chair–chair (molecule *B*) conformations (Hendrickson, 1967*a*), with bond angles designated as follows: θ_1 C2—C1—C8, C6—C5—C4; θ_2 C1—C8—O2, C5—C4—O1; θ_3 C8—O2—C7, C4—O1—C3; θ_4 O2—C7—C6, O1—C3—C2; θ_5 C7—C6—C5, C3—C2—C1. The values of these parameters are, for molecules *A* and *B* respectively: θ_1 = 119.0 (3), 116.6 (3)° and 119.3 (3), 118.9 (3)°; θ_2 = 111.9 (3), 105.5 (3)° and 108.2 (3), 108.0 (3)°; θ_3 = 115.6 (3), 116.4 (3)° and 114.2 (3), 115.1 (3)°; θ_4 = 107.5 (3), 110.8 (4)° and 115.9 (3), 111.4 (3)°; θ_5 = 118.2 (3), 119.6 (4)° and 119.5 (3), 119.2 (3)°. These values are distorted from the theoretical value (Hendrickson, 1967*b*) of the long-chair form, $\theta_1 = \theta_2 = \theta_3 = \theta_4 = \theta_5 = 118°$, and are distorted from the experimental values $\theta_1 = \theta_2 = \theta_5 = 118°$ and $\theta_3 = \theta_4 = 115°$ (Hendrickson, 1967*b*). Torsion-angle designations are: ω_1 C3—C2—C1—C8, C7—C6—C5—C4; ω_2 C2—C1—C8—O2, C6—C5—C4—O1; ω_3 C1—C8—O2—C7, C5—C4—O1—C3; ω_4 C8—O2—C7—C6, C4—O1—C3—C2; ω_5 O2—C7—C6—C5, O1—C3—C2—C1. The values for molecules *A* and *B* are respectively: ω_1 = 1.5 (6), 2.9 (5)° and 8.1 (5), 2.5 (5)°; ω_2 = 70.4 (5), 69.9 (4)° and −55.5 (4), 82.5 (4)°; ω_3 = −146.8 (4), −160.7 (3)° and 175.7 (3), −146.8 (3)°; ω_4 = 155.7 (3), 158.5 (3)° and −52.3 (4), 148.0 (3)°; ω_5 = −83.3 (4), −69.9 (5)° and −50.3 (5), −50.8 (5)°. These are distorted from those of the symmetrical long-chair conformation (of cyclodecane), having torsion angles of $\omega_1 = \pm 0$, $\omega_2 = \pm 112.2$, $\omega_3 = \pm 72.7$, $\omega_4 = \pm 72.7$ and $\omega_5 = \pm 112.2°$ (Hendrickson, 1967*a*).

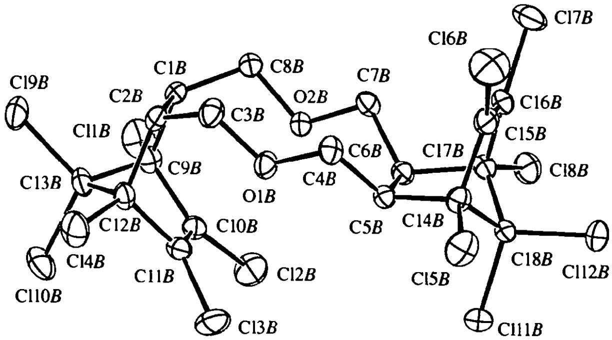


Fig. 1. One of the two independent molecules, with ellipsoids at the 30% probability level. H atoms have been omitted for clarity.

Experimental

Crystals suitable for analysis were prepared by reacting 1,6-dioxacyclodeca-3,8-diene (Reppe, 1955) with hexachlorocyclopentadiene, following a modification of the method of Akhtar *et al.* (1968). Crystals (m.p. 557–559 K) were grown by slow cooling of a *p*-xylene solution.

Crystal data

$C_{18}H_{12}Cl_{12}O_2$
$M_r = 685.73$
Monoclinic
$C2/c$
$a = 26.831\ (2)$ Å
$b = 15.2944\ (7)$ Å
$c = 24.952\ (1)$ Å
$\beta = 94.00\ (1)°$
$V = 10214\ (1)$ Å^3
$Z = 16$
$D_x = 1.783$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 5$–$11°$
$\mu = 1.33$ mm^{-1}
$T = 296$ K
Fragment
0.32 × 0.32 × 0.13 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scans (North *et al.*, 1968)
$T_{\min} = 0.74$, $T_{\max} = 0.84$

6584 reflections with $I > \sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 31$
$k = 0 \rightarrow 18$
$l = -29 \rightarrow 29$

9529 measured reflections
8968 independent reflections

3 standard reflections
frequency: 120 min
intensity decay: 0.2%

Refinement

Refinement on F
$R = 0.057$
$wR = 0.039$
$S = 1.243$
6584 reflections
674 parameters
H atoms refined isotropically
$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0004F_o^4]$
$(\Delta/\sigma)_{\max} = 0.028$

$\Delta\rho_{\max} = 0.44$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}
Extinction correction: isotropic (Zachariasen, 1963)
Extinction coefficient: $1.9(3) \times 10^{-8}$
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

O1A—C3A	1.419 (5)	O1B—C3B	1.397 (5)
O1A—C4A	1.416 (5)	O1B—C4B	1.417 (5)
O2A—C7A	1.423 (5)	O2B—C7B	1.413 (5)
O2A—C8A	1.424 (5)	O2B—C8B	1.417 (5)
C1A—C2A	1.577 (6)	C1B—C2B	1.576 (6)
C5A—C6A	1.585 (6)	C5B—C6B	1.576 (6)
C10A—C11A	1.314 (6)	C10B—C11B	1.309 (6)
C15A—C16A	1.305 (6)	C15B—C16B	1.318 (6)
C3A—O1A—C4A	115.1 (3)	C3B—O1B—C4B	116.4 (3)
C7A—O2A—C8A	114.2 (3)	C7B—O2B—C8B	115.6 (3)
Cl9A—C13A—Cl10A	107.2 (2)	Cl9B—C13B—Cl10B	107.3 (2)
Cl11A—C18A—Cl12A	108.3 (2)	Cl11B—C18B—Cl12B	107.6 (2)
C8A—C1A—C2A—C3A	8.1 (5)	C8B—C1B—C2B—C3B	1.5 (6)
C9A—C1A—C2A—C12A	1.0 (4)	C9B—C1B—C2B—C12B	1.3 (4)
C4A—C5A—C6A—C7A	2.5 (5)	C4B—C5B—C6B—C7B	2.9 (5)
C14A—C5A—C6A—C17A	0.2 (4)	C14B—C5B—C6B—C17B	1.0 (4)

C—H distances are in the range 0.89 (3)–1.03 (3) Å, while $B_{\rm iso}$ values for H atoms are in range the 1.0 (7)–6 (1) Å^2.

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Operations Manual*. Data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF IN* in *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1349). Services for accessing these data are described at the back of the journal.

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Adenosine-5′-carboxylic Acid

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Abstract

The two molecules in the asymmetric unit of adenosine-5′-carboxylic acid, $C_{10}H_{11}N_5O_5$, exist as zwitterions with N1 protonated and the carboxyl groups ionized. Both molecules are in an *anti* conformation with glycosyl torsion angles of −161.4 (3) and −155.5 (3)°. The ribose moieties adopt a C3-*endo*–C2-*exo* twist conformation. The pseudo-rotation parameters are P =

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