N2—C6—C7	114.0 (8)	C6—C7—C8	103.0 (8)
N2—C9—C8	112.9 (8)	C7—C8—C9	102.3 (8)
N3—C11—C12	107.5 (9)	C11—C12—C13	106.2 (8)
N3—C14—C13	107.0 (8)	C12—C13—C14	109.5 (8)
N4—C16—C17	114.2 (7)	C16—C17—C18	101.3 (7)
N4—C19—C18	114.5 (7)	C17—C18—C19	101.8 (7)
N1—C1—C20	126.4 (8)	C2—C1—C20	127.4 (8)
N1—C4—C5	128.4 (8)	C3—C4—C5	126.5 (9)
N2—C6—C5	125.2 (9)	C5—C6—C7	120.8 (9)
N2—C9—C10	122.9 (9)	C8—C9—C10	124.3 (8)
N3—C11—C10	121.9 (8)	C10—C11—C12	130.5 (8)
N3—C14—C15	137.6 (8)	C13—C14—C15	115.4 (8)
N4—C16—C15	118.9 (8)	C15—C16—C17	126.8 (8)
N4—C19—C20	123.0 (9)	C18—C19—C20	122.4 (8)

The data do not discriminate between enantiomorphs. The enantiomorph was chosen to be consistent with all other known chlorophyll and bacteriochlorophyll structures. The coordinates of N1 were fixed to define the origin.

Data collection: CAD-4 (Enraf-Nonius, 1977). Cell refinement: CAD-4. Data reduction: *MolEN* (Fair, 1990). Structure solution: *MULTAN*80 (Main *et al.*, 1980). Molecular graphics: *ORTEP*II (Johnson, 1976) and *GENPLOT* (Computer Graphics Service, 1989).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1097). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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8-Chloro-3,6-dioxaoctyl (*E*,*E*)-2-Chloro-3-(3,5-dichloro-2-hydroxyphenylazo)-2-butenoate

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Abstract

The title molecule, $C_{16}H_{18}Cl_4N_2O_5$, shows an *E*,*E* configuration. An intramolecular three-center hydrogen bond is formed and stacking between the molecules indicates the presence of weak π-electron interactions.

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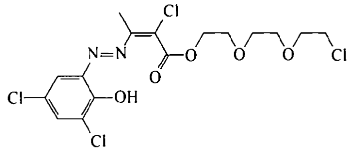
Comment

8-Chloro-3,6-dioxaoctyl 2-chloro-3-(3,5-dichloro-2-hydroxyphenylazo)-2-butenoate (I) was synthesized as a mixture of *E*,*Z* and *E*,*E* isomers and the catalytic and complexing properties were investigated as part of a research program involving new complexing agents. A satisfactory determination of the configurations of the isomers by usual NMR techniques was not possible. Therefore, the isomers were separated by column chromatography and their structures determined by X-ray structure analysis. The structure of the *E*,*E* isomer is reported here. An *ORTEP*II drawing (Johnson, 1971) of the molecule with the atomic numbering scheme is shown in Fig. 1.



(I)

The bond parameters are in good agreement with those of azoalkenes (Kirschke, Baumann, Costisella & Ramm, 1994). Bond lengths indicate N1═N2 and C7═C9 to be double bonds. The molecule shows an *E*,*E* configuration with an intramolecular three-center hydrogen bond. This hydrogen bond is characterized by the following geometry: (*a*) O1—H1 1.23 (5), O1···O2 2.800 (4), H1···O2 1.96 (5) Å and O1—H1···O2 121 (3)°; (*b*) O1···N2 2.575 (3), H1···N2 1.52 (5) Å and O1—H1···N2 138 (4)°. Similar three-center hydrogen bonds were found in other azoalkenes (Kirschke, Baumann, Costisella & Ramm, 1994). The packing of the molecules in the unit cell is shown in Fig. 2. The molecular arrangement is determined by van der Waals interactions and stacking between molecules (x, y, z) and ($1-x$, $-y$, $1-z$). The mean distance between the two molecules is 3.662 (4) Å, indicating weak π-electron interactions.

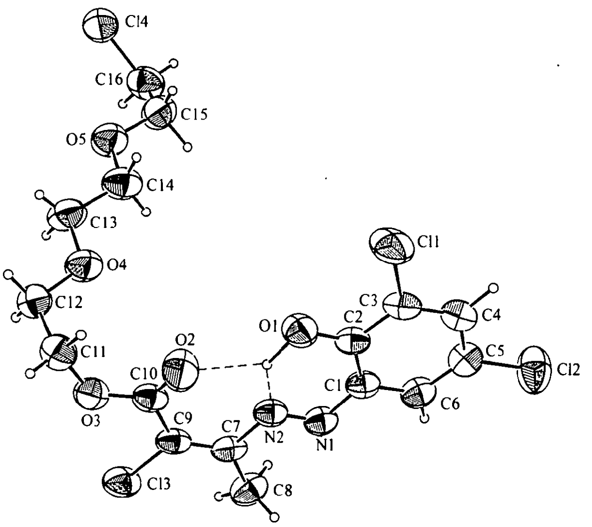


Fig. 1. An *ORTEP*II (Johnson, 1971) drawing of the molecular structure showing the crystallographic numbering scheme (50% probability displacement ellipsoids). The three-center hydrogen bond is shown by dashed lines.

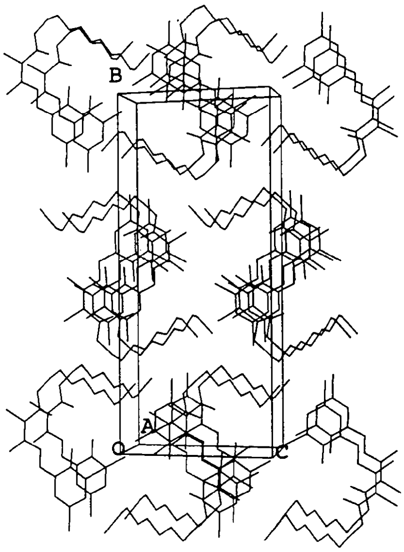


Fig. 2. A *PLUTO* (Motherwell & Clegg, 1976) drawing of the molecular packing in the title structure.

Experimental

Crystals were obtained by recrystallization of a mixture of dichloromethane/THF (9:1 *v*/*v*).

Crystal data

$C_{16}H_{18}Cl_4N_2O_5$
$M_r = 460.14$
Monoclinic
$P2_1/n$
$a = 7.1189$ (4) Å
$b = 25.685$ (3) Å
$c = 11.1476$ (9) Å
$\beta = 98.641$ (6)°
$V = 2015.2$ (3) Å^3
$Z = 4$
$D_x = 1.517$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 36$–$40°$
$\mu = 5.731$ mm^{-1}
$T = 293$ K
Plate
0.52 × 0.50 × 0.27 mm
Red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF
$T_{\min} = 0.745$, $T_{\max} = 1.498$
4068 measured reflections
3986 independent reflections

2758 observed reflections [$F > 3.0\sigma(F)$]
$R_{\rm int} = 0.035$
$\theta_{\max} = 75°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 31$
$l = -13 \rightarrow 13$
2 standard reflections
frequency: 60 min
intensity variation: −2.4%

Refinement

Refinement on *F*
$R = 0.045$
$wR = 0.071$
$S = 2.459$
2758 reflections
317 parameters
All H-atom parameters refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.02$
$\Delta\rho_{\max} = 0.342$ e Å^{-3}
$\Delta\rho_{\min} = -0.358$ e Å^{-3}
Extinction correction: Becker & Coppens type 1, isotropic
Extinction coefficient: 1.0166×10^{-6}
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)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$U_{\rm eq}$
Cl1	0.27730 (19)	0.02987 (4)	0.06612 (7)	0.0816 (4)
Cl2	0.27354 (19)	0.20683 (4)	0.32066 (9)	0.0862 (4)
Cl3	0.22017 (14)	−0.12438 (4)	0.77274 (7)	0.0671 (3)
Cl4	−0.94626 (16)	−0.17921 (4)	0.02035 (10)	0.0793 (3)
O1	0.2722 (4)	−0.02052 (9)	0.29773 (19)	0.0613 (8)
O2	0.2695 (5)	−0.11181 (11)	0.4347 (2)	0.0828 (12)
O3	0.1964 (3)	−0.17503 (9)	0.5537 (2)	0.0575 (8)
O4	−0.1038 (3)	−0.19167 (10)	0.3518 (2)	0.0644 (8)
O5	−0.5642 (4)	−0.16776 (10)	0.1887 (2)	0.0688 (9)
N1	0.2556 (4)	0.03340 (11)	0.5294 (2)	0.0503 (8)
N2	0.2508 (4)	−0.01559 (10)	0.5260 (2)	0.0476 (8)
C1	0.2648 (5)	0.05756 (13)	0.4170 (3)	0.0468 (9)
C2	0.2705 (5)	0.03084 (12)	0.3071 (3)	0.0468 (9)
C3	0.2774 (5)	0.06122 (13)	0.2034 (3)	0.0532 (10)
C4	0.2799 (5)	0.11463 (13)	0.2072 (3)	0.0547 (10)
C5	0.2742 (5)	0.13962 (13)	0.3167 (3)	0.0562 (10)
C6	0.2645 (5)	0.11164 (13)	0.4210 (3)	0.0538 (10)
C7	0.2414 (5)	−0.03989 (14)	0.6390 (3)	0.0500 (10)
C8	0.2355 (6)	−0.00657 (15)	0.7479 (3)	0.0664 (11)
C9	0.2349 (5)	−0.09207 (14)	0.6383 (3)	0.0496 (10)
C10	0.2361 (5)	−0.12654 (14)	0.5301 (3)	0.0510 (10)
C11	0.1946 (5)	−0.21376 (14)	0.4582 (3)	0.0601 (11)
C12	−0.0026 (5)	−0.23223 (13)	0.4179 (3)	0.0574 (10)
C13	−0.2902 (6)	−0.20558 (15)	0.2979 (4)	0.0691 (12)
C14	−0.3685 (6)	−0.15970 (17)	0.2271 (4)	0.0720 (14)
C15	−0.6513 (6)	−0.12308 (15)	0.1284 (4)	0.0702 (14)
C16	−0.8574 (6)	−0.12765 (16)	0.1198 (4)	0.0747 (14)

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

Cl1—C3	1.729 (3)	N2—C7	1.416 (4)
Cl2—C5	1.727 (3)	C1—C2	1.410 (5)
Cl3—C9	1.730 (4)	C1—C6	1.390 (5)
Cl4—C16	1.781 (4)	C2—C3	1.402 (5)
O1—C2	1.324 (4)	C3—C4	1.373 (5)
O2—C10	1.186 (4)	C4—C5	1.385 (5)
O3—C10	1.312 (4)	C5—C6	1.377 (5)
O3—C11	1.456 (4)	C7—C8	1.491 (5)
O4—C12	1.410 (4)	C7—C9	1.341 (5)
O4—C13	1.418 (5)	C9—C10	1.497 (5)
O5—C14	1.410 (5)	C11—C12	1.486 (5)
O5—C15	1.424 (5)	C13—C14	1.480 (6)
N1—N2	1.259 (4)	C15—C16	1.461 (6)
N1—C1	1.409 (4)		
C10—O3—C11	118.7 (3)	C4—C5—C6	120.9 (3)
C12—O4—C13	114.1 (3)	C1—C6—C5	119.6 (3)
C14—O5—C15	111.4 (3)	N2—C7—C8	118.8 (3)
N2—N1—C1	114.7 (2)	N2—C7—C9	116.2 (3)
N1—N2—C7	114.7 (2)	C8—C7—C9	125.0 (3)
N1—C1—C2	124.7 (3)	Cl3—C9—C7	118.7 (3)
N1—C1—C6	114.3 (3)	Cl3—C9—C10	115.0 (3)
C2—C1—C6	121.0 (3)	C7—C9—C10	126.2 (3)
O1—C2—C1	123.7 (3)	O2—C10—O3	124.1 (3)
O1—C2—C3	119.2 (3)	O2—C10—C9	124.0 (3)
C1—C2—C3	117.0 (3)	O3—C10—C9	111.8 (3)
Cl1—C3—C2	118.4 (3)	O3—C11—C12	110.1 (3)
Cl1—C3—C4	119.4 (3)	O4—C12—C11	107.9 (3)
C2—C3—C4	122.2 (3)	O4—C13—C14	106.2 (3)
C3—C4—C5	119.2 (3)	O5—C14—C13	108.7 (3)
Cl2—C5—C4	119.1 (3)	O5—C15—C16	109.2 (3)
Cl2—C5—C6	120.0 (3)	Cl4—C16—C15	111.1 (3)

Data were corrected for Lorentz and polarization effects, and for absorption using *DIFABS* (Walker & Stuart, 1983). H atoms were located by difference Fourier synthesis and refined isotropically.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN PROCESS* (Fair, 1990). Program(s) used to solve structure: *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Program(s) used to refine structure: *MolEN LSFM*. Software used to prepare material for publication: *PLATON* (Spek, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, and torsion angles have been deposited with the IUCr (Reference: SE1046). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-Azabicyclo[4.4.0]decane Borine Adduct

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Abstract

The title compound, (1-azoniabicyclo[4.4.0]decane)trihydroborate, $C_9H_{20}BN$, adopts a *trans*-decalin-type con-

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