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(1α,2β,3α,6β)-3,6-Dichlorocyclohex-4-ene-1,2-diyl diacetate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.049
wR factor = 0.145
Data-to-parameter ratio = 9.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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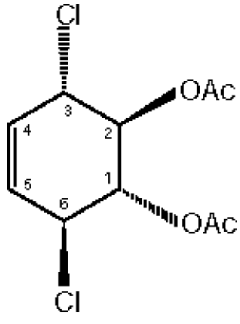
We present here a second positional isomer of $C_{10}H_{12}Cl_2O_4$. The crystal was found to be inversion twinned. The cyclohexene ring adopts a distorted half-chair conformation.

Comment

As part of an ongoing research program to design and synthesize novel haloconduritol compounds, we successfully used the diacetate and reported (Baran *et al.*, 2003, 2004) an efficient preparation of (1α,2α,3β,6β)-6-halocyclohex-4-ene-1,2,3-triols (halogen = Cl and Br).

Another isomer of $C_{10}H_{12}Cl_2O_4$ was published by Öztürk *et al.* (2004) with the Cl atoms at position 5 and 6 and the acetate groups at position 3 and 4 of the cyclohexene ring. The Cl—C and C—O bond lengths are in the ranges 1.816 (4)–1.831 (5) and 1.349 (5)–1.438 (5) Å, respectively. All bond distances and angles are normal with respect to literature values (Öztürk *et al.*, 2004; Allen *et al.*, 1987). The cyclohexene ring adopts a distorted half-chair conformation. The puckering parameters (Cremer & Pople, 1975) for the cyclohexene ring are Q = 0.511 (4) Å, θ = 49.2 (6)° and φ = 148.8 (7)°.



(I)

The crystal structure of the title compound, (I), is stabilized by C—H···O and C—H···Cl inter- and intramolecular hydrogen bonds, in addition to van der Waals interactions (Table 2 and Fig. 2).

Experimental

The title compound was prepared according to the method of Öztürk *et al.* (2004) (yield: 42%, m.p. 442–443 K). ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.81 (*s*, 2H, H_4 and H_5), 5.30–5.26 (*AA′* part of *AA′XX′* system, 2H, H_1 and H_2), 4.67–4.63 (*XX′* part of *AA′XX′* system, 2H, H_3 and H_6), 2.08 (*s*, 6H, 2 × CH_3). ^{13}C NMR (50 MHz, $CDCl_3$): δ 169.3 (2 × C═O), 128.4 (C_4 and C_5), 74.1 (C_1 and C_2), 56.5 (C_3 and C_6), 20.4 (2 × CH_3). Analysis calculated for $C_{10}H_{12}Cl_2O_4$: C 44.97, H 4.53%; found: C 44.89, H 4.55%.

Crystal data

$C_{10}H_{12}Cl_2O_4$
M_r = 267.10
Orthorhombic, $P2_12_12_1$
a = 7.8525 (7) Å
b = 7.8036 (5) Å
c = 20.4482 (12) Å
V = 1253.02 (16) Å^3
Z = 4
D_x = 1.416 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1448 reflections
θ = 2.0–26.3°
μ = 0.51 mm^{-1}
T = 293 K
Prism, colorless
0.45 × 0.41 × 0.38 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
17 519 measured reflections
1448 independent reflections
1248 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 26.0°
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.145
S = 1.05
1448 reflections
147 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.1053P)^2 + 0.107P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.035 (8)

Table 1
Selected geometric parameters (Å, °).

Cl1—C2	1.831 (5)	O2—C7	1.198 (5)
Cl2—C5	1.816 (4)	O3—C4	1.438 (5)
O1—C3	1.433 (5)	O3—C9	1.349 (5)
O1—C7	1.350 (4)	O4—C9	1.203 (5)
C3—O1—C7	119.2 (3)	Cl2—C5—C4	108.5 (3)
C4—O3—C9	117.5 (3)	Cl2—C5—C6	109.9 (3)
Cl1—C2—C1	108.9 (3)	O1—C7—O2	123.2 (4)
Cl1—C2—C3	108.5 (3)	O1—C7—C8	110.5 (3)
O1—C3—C2	108.4 (3)	O2—C7—C8	126.2 (4)
O1—C3—C4	107.6 (3)	O3—C9—O4	123.4 (4)
O3—C4—C3	108.3 (3)	O3—C9—C10	110.0 (4)
O3—C4—C5	108.9 (3)	O4—C9—C10	126.6 (4)
C3—O1—C7—C8	173.1 (4)	Cl1—C2—C3—O1	−76.0 (3)
C3—O1—C7—O2	−3.5 (7)	C2—C3—C4—C5	−64.1 (4)
C4—O3—C9—C10	−176.8 (4)	O1—C3—C4—O3	60.0 (4)
C4—O3—C9—O4	3.3 (6)	O3—C4—C5—Cl2	−71.9 (3)
C6—C1—C2—Cl1	−136.8 (4)	C3—C4—C5—Cl2	169.6 (2)
Cl1—C2—C3—C4	167.3 (3)	Cl2—C5—C6—C1	−137.5 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2	0.98	2.31	2.718 (5)	104
C4—H4···O4	0.98	2.25	2.688 (5)	106
C6—H6···O4^{i}	0.93	2.52	3.364 (6)	151
C8—H8B···O2^{ii}	0.96	2.43	3.388 (7)	174
C8—H8C···Cl1^{iii}	0.96	2.70	3.591 (5)	155

Symmetry codes: (i) $x, y-1, z$; (ii) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iii) $-x, \frac{1}{2}+y, \frac{3}{2}-z$.

All H atoms were positioned geometrically (C—H = 0.9294–0.9807 Å) and refined with a riding model, with U_{iso} values constrained to be 1.2 (1.5 for methyl groups) times U_{eq} of the parent atom. The crystal under investigation was an inversion twin with a ratio of 0.45 (17):0.55 (17).

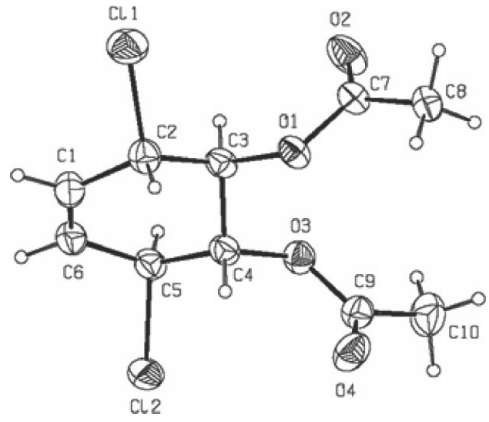


Figure 1
An *ORTEP*-3 drawing (Farrugia, 1997) of the title compound, with the atom-numbering scheme and 20% probability displacement ellipsoids.

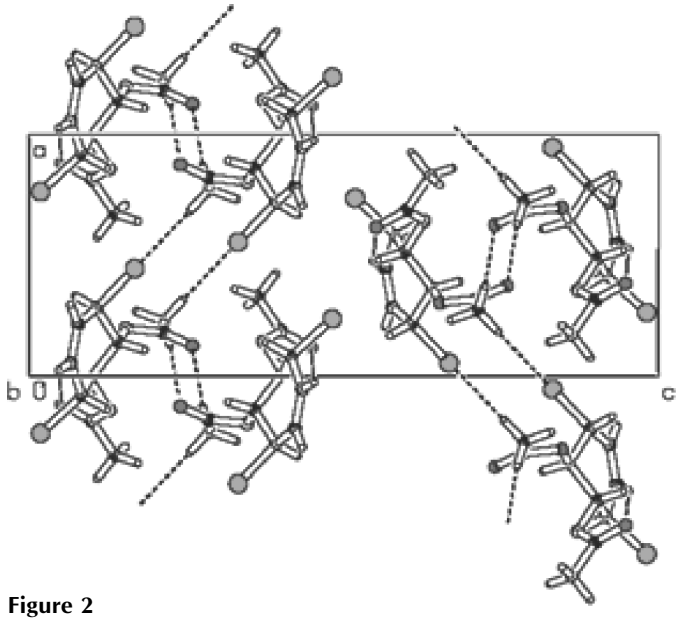


Figure 2
A packing diagram for (I), with hydrogen bonds shown as dashed lines. The view is down the b axis.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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