

**(1 $\alpha$ ,2 $\beta$ ,3 $\alpha$ ,6 $\beta$ )-3,6-Dichlorocyclohex-4-ene-1,2-diyl diacetate**Mehmet Akkurt,<sup>a</sup> Sema Öztürk,<sup>a\*</sup> Arif Baran,<sup>b</sup> Hasan Seçen<sup>b</sup> and Orhan Büyükgüngör<sup>c</sup><sup>a</sup>Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, <sup>b</sup>Department of Chemistry, Faculty of Arts and Sciences, Atatürk University, 25240 Erzurum, Turkey, and <sup>c</sup>Department of Physics, Ondokuz Mayıs University, 55139 Samsun, Turkey

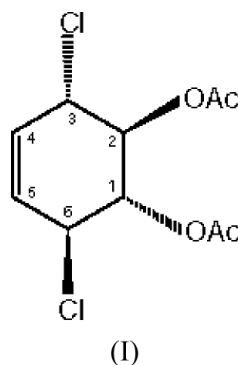
Correspondence e-mail: ozturk@erciyes.edu.tr

**Key indicators**Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å  
 $R$  factor = 0.049  
 $wR$  factor = 0.145  
Data-to-parameter ratio = 9.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.We present here a second positional isomer of  $\text{C}_{10}\text{H}_{12}\text{Cl}_2\text{O}_4$ . The crystal was found to be inversion twinned. The cyclohexene ring adopts a distorted half-chair conformation.

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**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 (1 $\alpha$ ,2 $\alpha$ ,3 $\beta$ ,6 $\beta$ )-6-halocyclohex-4-ene-1,2,3-triols (halogen = Cl and Br).Another isomer of  $\text{C}_{10}\text{H}_{12}\text{Cl}_2\text{O}_4$  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,  $\theta = 49.2$  (6)° and  $\varphi = 148.8$  (7)°.

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). <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  5.81 (s, 2H, H<sub>4</sub> and H<sub>5</sub>), 5.30–5.26 (AA' part of AA'XX' system, 2H, H<sub>1</sub> and H<sub>2</sub>), 4.67–4.63 (XX' part of AA'XX' system, 2H, H<sub>3</sub> and H<sub>6</sub>), 2.08 (s, 6H, 2 × CH<sub>3</sub>). <sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>):  $\delta$  169.3 (2 × C=O), 128.4 (C<sub>4</sub> and C<sub>5</sub>), 74.1 (C<sub>1</sub> and C<sub>2</sub>), 56.5 (C<sub>3</sub> and C<sub>6</sub>), 20.4 (2 × CH<sub>3</sub>). Analysis calculated for C<sub>10</sub>H<sub>12</sub>Cl<sub>2</sub>O<sub>4</sub>: 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<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.416$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 1448 reflections  
 $\theta = 2.0$ – $26.3^\circ$   
 $\mu = 0.51$  mm<sup>-1</sup>  
 $T = 293$  K  
 Prism, colorless  
 $0.45 \times 0.41 \times 0.38$  mm

Data collection

Stoe IPDS-II diffractometer  
 $\omega$  scans  
 17 519 measured reflections  
 1448 independent reflections  
 1248 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.056$   
 $\theta_{max} = 26.0^\circ$   
 $h = -9 \rightarrow 9$   
 $k = -9 \rightarrow 9$   
 $l = -25 \rightarrow 25$

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<sup>-3</sup>  
 $\Delta\rho_{min} = -0.21$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.035 (8)

Table 1

Selected geometric parameters (Å, °).

C11–C2	1.831 (5)	O2–C7	1.198 (5)
C12–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)	C12–C5–C4	108.5 (3)
C4–O3–C9	117.5 (3)	C12–C5–C6	109.9 (3)
C11–C2–C1	108.9 (3)	O1–C7–O2	123.2 (4)
C11–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)	C11–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–C12	–71.9 (3)
C6–C1–C2–C11	–136.8 (4)	C3–C4–C5–C12	169.6 (2)
C11–C2–C3–C4	167.3 (3)	C12–C5–C6–C1	–137.5 (4)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3–H3 <sup>ii</sup> ⋯O2	0.98	2.31	2.718 (5)	104
C4–H4 <sup>ii</sup> ⋯O4	0.98	2.25	2.688 (5)	106
C6–H6 <sup>ii</sup> ⋯O4 <sup>i</sup>	0.93	2.52	3.364 (6)	151
C8–H8B <sup>ii</sup> ⋯O2 <sup>iii</sup>	0.96	2.43	3.388 (7)	174
C8–H8C <sup>ii</sup> ⋯Cl1 <sup>iii</sup>	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).

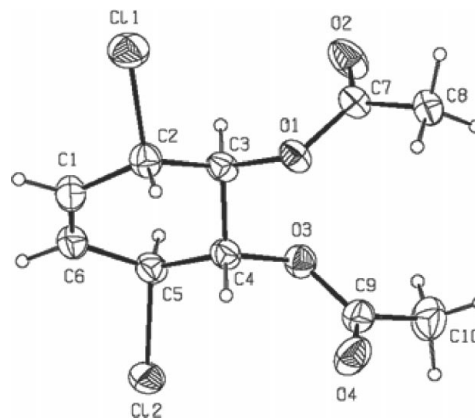


Figure 1

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

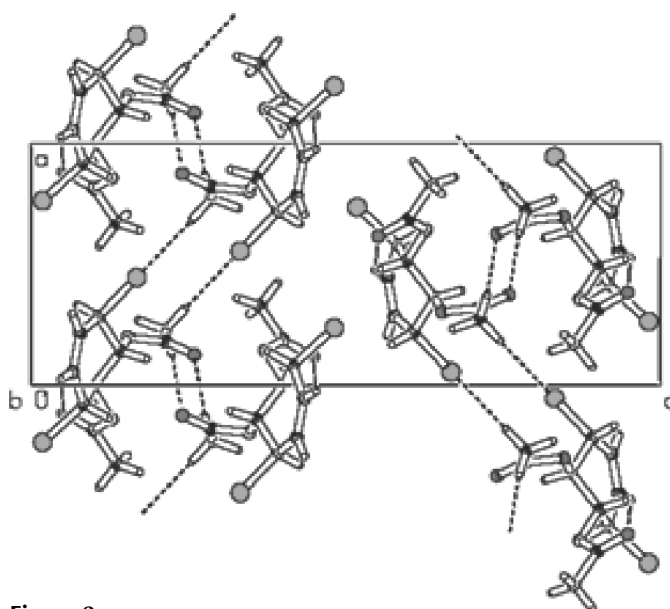


Figure 2

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-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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