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1,4-Bis(dibromomethylidene)cyclohexane: crystal packing with Br···Br and Br···π contacts

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(Wae) = 0.011 Å
R factor = 0.041
wR factor = 0.105
Data-to-parameter ratio = 16.1

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

The title compound, $C_8H_8Br_4$, displays crystallographic inversion symmetry. The packing involves interpenetrating corrugated layers built up *via* Br···Br and Br···π contacts.

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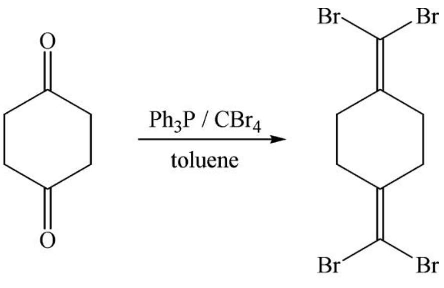
Comment

The title compound, (2), was synthesized as part of a study of compounds related to 7,7,8,8-tetracyano-*p*-quinodimethane (TCNQ; Hopf *et al.*, 2002). We present here its structure. The molecule, which possesses crystallographic inversion symmetry, is shown in Fig. 1. Bond lengths and angles may be regarded as normal. The double-bond length of 1.316 (11) Å may be compared to the 1.321 Å (no s.u. available) in 4-dichoromethylidene-perhydropyran-2-one (Dillon *et al.*, 1997), the only other structure with a $Hal_2C{=}C(CH_2)_2$ unit found in a search of the Cambridge Structural Database (Version 5.26; Allen, 2002). The six atoms of the double-bond system are coplanar, with an r.m.s. deviation of 0.028 Å. The Br—C—Br angle is narrow at 111.7 (4)°, which is, however, normal for $Hal_2C{=}C$ fragments; the mean value for 161 such fragments in the Cambridge Structural Database is 112.8° (search conditions: no disorder, only organics). The ring displays a nearly ideal chair conformation.



(1)
(2) (40%)

The crystal packing (Fig. 2) is based on two types of short contact, Br···Br and Br···π; there are no short H···Br contacts. The contact Br2···Br2($-x$, $1 - y$, $1 - z$) measures 3.496 (2) Å. The associated C—Br···Br angles are equal by symmetry at 152.0 (2)°, thus corresponding to a type I contact (Pedireddi *et al.*, 1994). The next shortest Br···Br contact is Br1···Br2($\frac{1}{2} + x$, $\frac{1}{2} - y$, $\frac{1}{2} + z$) of 3.905 (1) Å, considerably in excess of the double van der Waals radius. The same symmetry operator is involved in the contact C1—Br1···(C1=C2), in which the C1—Br1 vector makes an angle of 84.4° with the double-bond plane. The contact distances from Br1 (Å) are 3.396 (7) to C1, 3.405 (7) to C2 and 3.34 to the bond mid-point. These distances may be compared to 3.18–3.36 Å established for Br···π in the metastable charge-transfer complexes of bromine with benzene and toluene (Vasilyev *et al.*, 2002).

The overall effects of the contacts is to form corrugated layers parallel to (011). The layers are interpenetrating in pairs; one layer occupies the voids of the other. Only one such layer is shown in Fig. 2.

Experimental

Compound (2) was prepared as described by Hopf *et al.* (2002) and recrystallized from dichloromethane–pentane. Spectroscopic and analytical data were consistent with the proposed structure.

Crystal data

$C_8H_8Br_4$
$M_r = 423.78$
Monoclinic, $P2_1/n$
$a = 6.6033\ (10)$ Å
$b = 12.3957\ (18)$ Å
$c = 6.7152\ (10)$ Å
$\beta = 102.783\ (12)°$
$V = 536.03\ (14)$ Å^3
$Z = 2$
$D_x = 2.626$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 62 reflections
$\theta = 3.5–12.5°$
$\mu = 14.96$ mm^{-1}
$T = 173\ (2)$ K
Tablet, colourless
0.4 × 0.3 × 0.15 mm

Data collection

Siemens P4 diffractometer
ω scans
Absorption correction: ψ scan (*XEMP*; Siemens, 1994)
$T_{\min} = 0.30$, $T_{\max} = 0.95$
973 measured reflections
901 independent reflections
738 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.076$
$\theta_{\max} = 25.0°$
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 7$
3 standard reflections every 247 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.105$
$S = 1.13$
901 reflections
56 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.064P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.96$ e Å^{-3}
$\Delta\rho_{\min} = -0.71$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.043 (4)

Table 1
Selected geometric parameters (Å, °).

Br1—C1	1.899 (7)	C1—C2	1.316 (11)
Br2—C1	1.894 (7)		
C2—C1—Br2	124.1 (6)	C1—C2—C3	123.7 (7)
C2—C1—Br1	124.2 (6)	C1—C2—C4$^{\rm i}$	123.0 (7)
Br2—C1—Br1	111.7 (4)	C3—C2—C4$^{\rm i}$	113.2 (6)
C4$^{\rm i}$—C2—C3—C4	−51.4 (10)	C3—C4—C2—C3$^{\rm i}$	−51.3 (10)
C2—C3—C4—C2$^{\rm i}$	50.9 (10)		

Symmetry codes: (i) $-x+1, -y+1, -z$.

H atoms were included using a riding model with fixed C—H bond lengths of 0.98 Å; $U_{\rm iso}$(H) values were fixed at $1.2U_{\rm eq}$ of the parent atom.

Data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP5* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

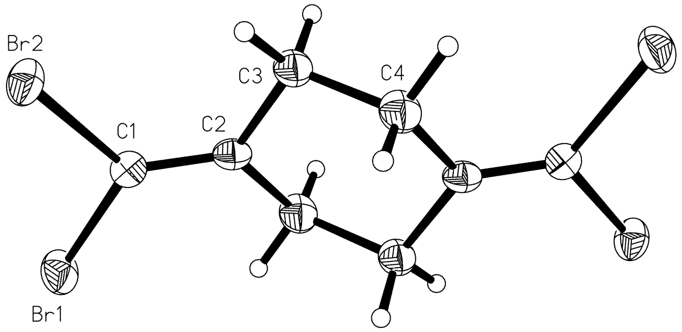


Figure 1
The molecule of the title compound in the crystal structure. Displacement ellipsoids are drawn at the 30% probability level. Unlabelled atoms are related to labelled atoms by $1-x, 1-y, -z$.

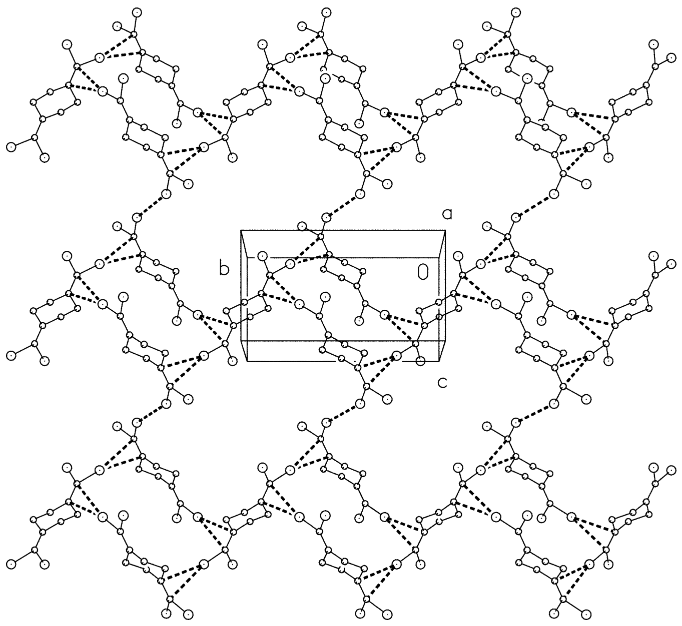


Figure 2
Packing diagram of the title compound at $x \simeq 0$ viewed perpendicular to the *bc* plane. Short contacts (see Comment) are indicated by dashed bonds. H atoms have been omitted.

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