Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

(1*RS*,2*SR*)-1,2,4,5,7-Pentabromo-5-methoxyindane

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
R factor = 0.041
wR factor = 0.073
Data-to-parameter ratio = 24.4

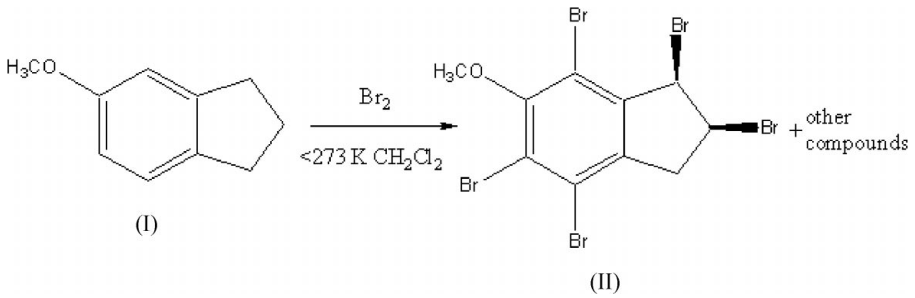
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 4 January 2005
Accepted 12 January 2005
Online 29 January 2005

In the title compound, $C_{10}H_7Br_5O$, prepared by bromination of 5-methoxyindane, all bond lengths and angles are in the usual ranges. However, the relatively wide range of Br—C—C angles [107.2 (5)–117.0 (4)°] in the five-membered ring may indicate repulsion between the neighbouring Br atoms. The crystal packing is stabilized by van der Waals interactions.

Comment

Indanes are present in a large number of natural products and in compounds of pharmaceutical importance (Quiclet-Sire *et al.*, 1999, and references therein). Indane-derived chiral ligands have also found application in transition metal-catalyzed processes (Zhang *et al.*, 2003, and references therein). Bromination of hydrocarbons often leads to useful intermediates used in the synthesis of bromoorganic compounds. These materials have numerous industrial applications as pesticides, plastics, fire retardants and pharmaceutical chemicals (Hileman, 1993). Bromoindanes are important key intermediates in the industrial and laboratory preparation of hydroxy and epoxide compounds (Crosman *et al.*, 2004), and of indenone and fluorenone compounds (Tutar *et al.*, 2001).



In the title compound, (II) (Fig. 1), prepared by bromination of 5-methoxyindane, (I), all bond lengths and angles (Table 1) are in the usual ranges observed in related structures (Allen *et al.*, 1987; Çelik *et al.*, 2002; Hökelek *et al.*, 1990, 1991, 1998; Akkurt *et al.*, 2004). The five-membered ring adopts the envelope conformation, with puckering parameters (Cremer & Pople, 1975) Q_2 = 0.309 (8) Å and φ_2 = 70.5 (15)°. The five Br—C distances in (II) range from 1.884 (7) to 1.980 (7) Å. The relatively wide range of Br—C—C angles [107.2 (5)–117.0 (4)°] in the five-membered ring may indicate repulsion between the neighbouring Br atoms.

There are no unusual short contacts between the molecules in (II). The crystal packing (Fig. 2) is stabilized by van der Waals interactions.

doi:10.1107/S160053680500111X

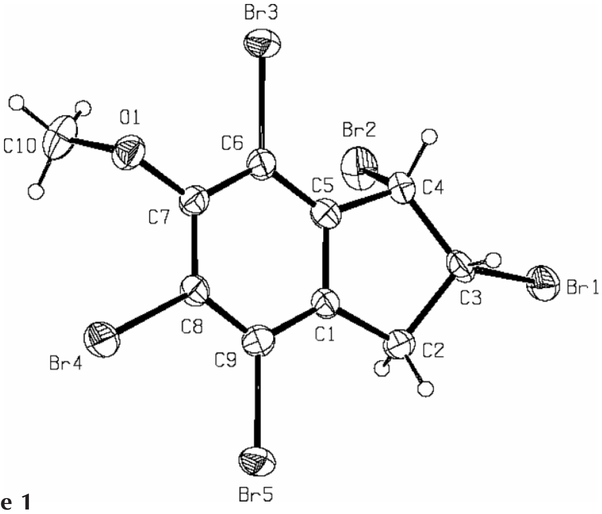


Figure 1
The molecular structure of (II). Displacement ellipsoids are drawn at the 50% probability level.

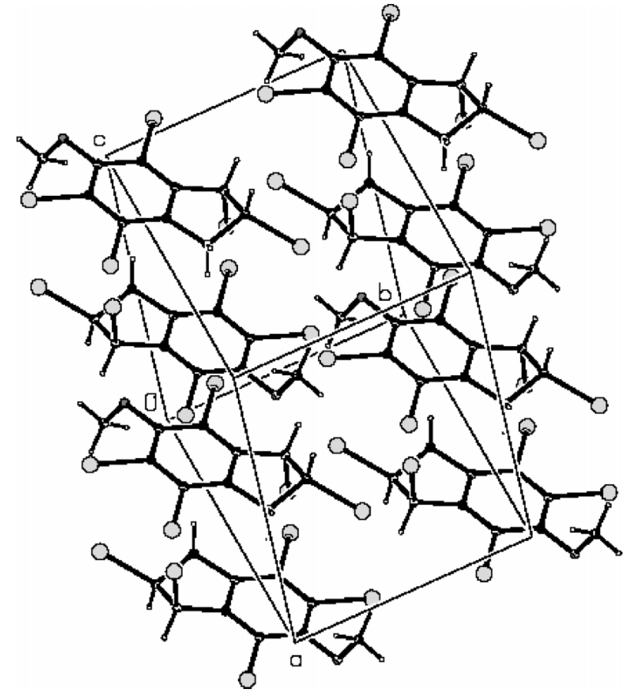


Figure 2
Packing diagram of (II), viewed approximately along [101].

Experimental

The title compound was prepared by bromination of 5-methoxyindane according to the following procedure. An excess of bromine was added to a solution of 5-methoxyindane in CH_2Cl_2 at below 273 K and allowed to stand for three weeks in a refrigerator. The reaction progress was monitored by thin-layer chromatography. After consumption of 5-methoxyindane, the excess bromine and solvent were removed at reduced pressure and at 293 K. The residue was crystallized from hexane–dichloromethane (1:5, 12 ml) at room temperature over a period of 1 d to give colourless block-like crystals (yield: 18%, m.p. 458–460 K).

Crystal data

$C_{10}H_7Br_5O$
$M_r = 542.66$
Triclinic, $P\overline{1}$
$a = 8.8895$ (11) Å
$b = 8.9281$ (10) Å
$c = 9.6787$ (13) Å
$\alpha = 98.382$ (10)°
$\beta = 113.160$ (10)°
$\gamma = 101.060$ (9)°
$V = 671.99$ (16) Å^3
$Z = 2$
$D_x = 2.682$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7984 reflections
$\theta = 2.4$–29.5°
$\mu = 14.93$ mm^{-1}
$T = 293$ K
Block, colourless
0.55 × 0.41 × 0.26 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED32*; Stoe & Cie, 2002)
$T_{\min} = 0.045$, $T_{\max} = 0.112$
13 393 measured reflections
3563 independent reflections
1562 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.167$
$\theta_{\max} = 29.6°$
$h = -12 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.074$
$S = 0.81$
3563 reflections
146 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0213P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.49$ e Å^{-3}
$\Delta\rho_{\min} = -0.67$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0076 (5)

Table 1
Selected geometric parameters (Å, °).

Br1—C3	1.930 (7)	Br5—C9	1.884 (7)
Br2—C4	1.980 (7)	O1—C7	1.368 (9)
Br3—C6	1.889 (7)	O1—C10	1.445 (10)
Br4—C8	1.889 (6)		
C7—O1—C10	112.5 (6)	O1—C7—C6	122.2 (6)
Br1—C3—C2	112.9 (5)	O1—C7—C8	120.3 (6)
Br1—C3—C4	117.0 (4)	Br4—C8—C7	117.2 (5)
Br2—C4—C3	112.6 (5)	Br4—C8—C9	120.8 (5)
Br2—C4—C5	107.2 (5)	Br5—C9—C1	118.1 (6)
Br3—C6—C5	121.1 (5)	Br5—C9—C8	122.2 (5)
Br3—C6—C7	117.9 (6)		

H atoms were geometrically positioned and refined with fixed individual displacement parameters [$U_{\rm iso}({\rm H}) = 1.2$–$1.5U_{\rm eq}({\rm C})$] using a riding model, with C—H = 0.98 (CH), 0.97 (CH_2) and 0.96 Å (CH_3).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); 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).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS-II diffractometer (purchased under grant F.279 of the University Research Fund). The authors are indebted to the Department of Chemistry (Gaziosmanpaşa University) for financial support (grant Nos. 2001/30 and 2002/12).

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supporting information

Acta Cryst. (2005). E**61**, o475–o477   [https://doi.org/10.1107/S160053680500111X]

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Crystal data

$C_{10}H_7Br_5O$
$M_r = 542.66$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.8895$ (11) Å
$b = 8.9281$ (10) Å
$c = 9.6787$ (13) Å
$\alpha = 98.382$ (10)°
$\beta = 113.16$ (1)°
$\gamma = 101.060$ (9)°
$V = 671.99$ (16) Å^3
$Z = 2$
$F(000) = 500$
$D_x = 2.682$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7984 reflections
$\theta = 2.4$–29.5°
$\mu = 14.93$ mm^{-1}
$T = 293$ K
Prism, colorless
0.55 × 0.41 × 0.26 mm

Data collection

Stoe IPDS-II diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
ω scans
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)
$T_{min} = 0.045$, $T_{max} = 0.112$
13393 measured reflections
3563 independent reflections
1562 reflections with $I > 2\sigma(I)$
$R_{int} = 0.167$
$\theta_{max} = 29.6°$, $\theta_{min} = 2.4°$
$h = -12 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.074$
$S = 0.81$
3563 reflections
146 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0213P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.67$ e Å^{-3}
Extinction correction: SHELXL97, $FC^* = KFC[1+0.001XFC^2\Lambda^3/SIN(2\Theta)]^{-1/4}$
Extinction coefficient: 0.0076 (5)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
Br1	0.05564 (9)	−0.38956 (7)	0.70585 (10)	0.0540 (3)
Br2	0.48174 (10)	−0.24854 (9)	0.93215 (10)	0.0616 (3)
Br3	0.77520 (9)	−0.15008 (8)	0.73156 (10)	0.0546 (3)
Br4	0.64356 (9)	0.43723 (8)	0.69282 (11)	0.0603 (3)
Br5	0.25845 (9)	0.29516 (8)	0.67368 (9)	0.0525 (3)
O1	0.8384 (5)	0.1947 (5)	0.7215 (6)	0.0494 (16)
C1	0.3545 (8)	0.0127 (7)	0.6895 (8)	0.0418 (19)
C2	0.1932 (8)	−0.0738 (7)	0.6881 (10)	0.050 (3)
C3	0.1973 (8)	−0.2469 (6)	0.6457 (8)	0.043 (2)
C4	0.3860 (7)	−0.2412 (7)	0.7115 (8)	0.043 (2)
C5	0.4666 (8)	−0.0819 (7)	0.7046 (8)	0.041 (2)
C6	0.6250 (8)	−0.0227 (7)	0.7149 (8)	0.0427 (19)
C7	0.6799 (7)	0.1324 (7)	0.7106 (8)	0.0401 (19)
C8	0.5661 (7)	0.2256 (6)	0.6950 (8)	0.043 (2)
C9	0.4079 (8)	0.1684 (7)	0.6862 (8)	0.045 (2)
C10	0.9656 (9)	0.2549 (10)	0.8803 (10)	0.066 (3)
H2A	0.09510	−0.05340	0.61070	0.0600*
H2B	0.19300	−0.04700	0.78890	0.0600*
H3	0.15150	−0.27910	0.53250	0.0520*
H4	0.40640	−0.32390	0.64820	0.0520*
H10A	1.07460	0.29540	0.88210	0.0990*
H10B	0.97000	0.17150	0.93310	0.0990*
H10C	0.93660	0.33760	0.93130	0.0990*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0565 (4)	0.0372 (4)	0.0754 (6)	0.0098 (3)	0.0356 (4)	0.0184 (4)
Br2	0.0694 (5)	0.0547 (4)	0.0508 (5)	0.0105 (4)	0.0158 (4)	0.0224 (4)
Br3	0.0496 (4)	0.0418 (4)	0.0786 (6)	0.0219 (3)	0.0282 (4)	0.0192 (4)
Br4	0.0586 (4)	0.0360 (4)	0.0943 (6)	0.0128 (3)	0.0370 (4)	0.0285 (4)
Br5	0.0521 (4)	0.0407 (3)	0.0716 (6)	0.0224 (3)	0.0274 (4)	0.0192 (3)
O1	0.040 (2)	0.050 (3)	0.058 (3)	0.009 (2)	0.021 (2)	0.018 (2)
C1	0.038 (3)	0.031 (3)	0.047 (4)	0.009 (3)	0.010 (3)	0.007 (3)
C2	0.038 (4)	0.034 (3)	0.082 (6)	0.011 (3)	0.027 (4)	0.020 (3)
C3	0.050 (4)	0.023 (3)	0.046 (4)	0.009 (3)	0.012 (3)	0.005 (3)

C4	0.040 (4)	0.032 (3)	0.047 (4)	0.006 (3)	0.013 (3)	0.001 (3)
C5	0.043 (4)	0.033 (3)	0.048 (4)	0.011 (3)	0.019 (3)	0.012 (3)
C6	0.038 (3)	0.035 (3)	0.044 (4)	0.010 (3)	0.007 (3)	0.009 (3)
C7	0.034 (3)	0.039 (3)	0.045 (4)	0.010 (3)	0.014 (3)	0.013 (3)
C8	0.040 (3)	0.027 (3)	0.060 (5)	0.008 (3)	0.017 (3)	0.021 (3)
C9	0.042 (4)	0.044 (3)	0.043 (4)	0.015 (3)	0.011 (3)	0.013 (3)
C10	0.041 (4)	0.082 (6)	0.053 (5)	0.002 (4)	0.007 (4)	0.010 (4)

Geometric parameters (Å, º)

Br1—C3	1.930 (7)	C4—C5	1.488 (9)
Br2—C4	1.980 (7)	C5—C6	1.364 (11)
Br3—C6	1.889 (7)	C6—C7	1.390 (9)
Br4—C8	1.889 (6)	C7—C8	1.406 (9)
Br5—C9	1.884 (7)	C8—C9	1.365 (10)
O1—C7	1.368 (9)	C2—H2A	0.9700
O1—C10	1.445 (10)	C2—H2B	0.9700
C1—C2	1.486 (11)	C3—H3	0.9800
C1—C5	1.404 (10)	C4—H4	0.9800
C1—C9	1.389 (9)	C10—H10A	0.9600
C2—C3	1.551 (9)	C10—H10B	0.9600
C3—C4	1.530 (10)	C10—H10C	0.9600
Br1···Br2	3.4015 (14)	Br3···H4	3.0700
Br1···Br3^{i}	3.6368 (13)	Br4···H4^{vii}	3.1300
Br1···Br4^{ii}	3.6377 (13)	Br4···H10C	3.1100
Br1···Br5^{iii}	3.6611 (12)	Br4···H4^{viii}	3.2300
Br1···Br1^{iv}	3.8217 (13)	Br5···H10A^{i}	3.0600
Br2···Br5^{v}	3.7367 (13)	Br5···H2A	3.0300
Br2···Br1	3.4015 (14)	O1···Br3	3.044 (5)
Br2···Br3	3.8664 (14)	O1···Br4	3.000 (5)
Br2···C1^{v}	3.514 (7)	O1···H3^{vii}	2.7000
Br2···C9^{v}	3.351 (7)	C1···Br2^{v}	3.514 (7)
Br3···Br2	3.8664 (14)	C9···Br3^{vii}	3.682 (7)
Br3···Br1^{vi}	3.6368 (13)	C9···Br2^{v}	3.351 (7)
Br3···C9^{vii}	3.682 (7)	C10···Br3	3.497 (9)
Br3···Br4^{iii}	3.5614 (12)	C10···Br4	3.564 (9)
Br3···O1	3.044 (5)	C6···H10B	2.9400
Br3···C10	3.497 (9)	C8···H10C	3.0400
Br3···Br5^{vii}	3.8208 (13)	H2A···Br5	3.0300
Br4···Br5	3.3361 (13)	H2A···H2A^{xi}	2.5900
Br4···O1	3.000 (5)	H3···Br1^{iv}	3.1700
Br4···Br3^{viii}	3.5614 (12)	H3···O1^{vii}	2.7000
Br4···Br1^{ix}	3.6377 (13)	H4···Br3	3.0700
Br4···C10	3.564 (9)	H4···Br4^{iii}	3.2300
Br5···Br3^{vii}	3.8208 (13)	H4···Br4^{vii}	3.1300
Br5···Br4	3.3361 (13)	H10A···Br5^{vi}	3.0600
Br5···Br2^{v}	3.7367 (13)	H10B···Br3	2.9700

Br5···Br1^{viii}	3.6611 (12)	H10B···C6	2.9400
Br1···H3^{iv}	3.1700	H10B···Br3^{x}	3.2000
Br3···H10B	2.9700	H10C···Br4	3.1100
Br3···H10B^{x}	3.2000	H10C···C8	3.0400

C7—O1—C10	112.5 (6)	C7—C8—C9	122.0 (6)
C2—C1—C5	111.4 (6)	Br5—C9—C1	118.1 (6)
C2—C1—C9	129.6 (7)	Br5—C9—C8	122.2 (5)
C5—C1—C9	118.9 (7)	C1—C9—C8	119.7 (7)
C1—C2—C3	101.2 (6)	C1—C2—H2A	112.00
Br1—C3—C2	112.9 (5)	C1—C2—H2B	112.00
Br1—C3—C4	117.0 (4)	C3—C2—H2A	112.00
C2—C3—C4	105.3 (5)	C3—C2—H2B	112.00
Br2—C4—C3	112.6 (5)	H2A—C2—H2B	109.00
Br2—C4—C5	107.2 (5)	Br1—C3—H3	107.00
C3—C4—C5	102.7 (5)	C2—C3—H3	107.00
C1—C5—C4	109.8 (6)	C4—C3—H3	107.00
C1—C5—C6	120.8 (6)	Br2—C4—H4	111.00
C4—C5—C6	129.5 (6)	C3—C4—H4	111.00
Br3—C6—C5	121.1 (5)	C5—C4—H4	111.00
Br3—C6—C7	117.9 (6)	O1—C10—H10A	109.00
C5—C6—C7	121.1 (7)	O1—C10—H10B	109.00
O1—C7—C6	122.2 (6)	O1—C10—H10C	109.00
O1—C7—C8	120.3 (6)	H10A—C10—H10B	109.00
C6—C7—C8	117.5 (6)	H10A—C10—H10C	110.00
Br4—C8—C7	117.2 (5)	H10B—C10—H10C	109.00
Br4—C8—C9	120.8 (5)		

C10—O1—C7—C8	93.8 (8)	Br2—C4—C5—C1	−100.8 (6)
C10—O1—C7—C6	−85.9 (8)	C3—C4—C5—C6	−163.8 (7)
C9—C1—C2—C3	164.3 (7)	C3—C4—C5—C1	18.1 (7)
C2—C1—C5—C4	1.1 (8)	C1—C5—C6—Br3	−178.9 (5)
C5—C1—C2—C3	−19.4 (8)	C4—C5—C6—C7	−178.0 (7)
C5—C1—C9—Br5	−177.7 (5)	C1—C5—C6—C7	−0.1 (11)
C5—C1—C9—C8	1.1 (10)	C4—C5—C6—Br3	3.2 (11)
C2—C1—C5—C6	−177.2 (7)	Br3—C6—C7—O1	−1.5 (9)
C9—C1—C5—C4	177.9 (6)	C5—C6—C7—C8	−0.1 (10)
C9—C1—C5—C6	−0.4 (10)	Br3—C6—C7—C8	178.7 (5)
C2—C1—C9—Br5	−1.5 (11)	C5—C6—C7—O1	179.7 (6)
C2—C1—C9—C8	177.2 (7)	O1—C7—C8—Br4	−0.8 (9)
C1—C2—C3—Br1	158.5 (5)	O1—C7—C8—C9	−179.0 (6)
C1—C2—C3—C4	29.8 (7)	C6—C7—C8—Br4	179.0 (5)
Br1—C3—C4—Br2	−40.8 (6)	C6—C7—C8—C9	0.8 (10)
C2—C3—C4—C5	−29.6 (7)	C7—C8—C9—C1	−1.3 (11)
C2—C3—C4—Br2	85.4 (6)	Br4—C8—C9—Br5	−0.7 (8)

Br1—C3—C4—C5	−155.8 (5)	Br4—C8—C9—C1	−179.4 (5)
Br2—C4—C5—C6	77.3 (8)	C7—C8—C9—Br5	177.4 (5)

Symmetry codes: (i) $x-1, y, z$; (ii) $x-1, y-1, z$; (iii) $x, y-1, z$; (iv) $-x, -y-1, -z+1$; (v) $-x+1, -y, -z+2$; (vi) $x+1, y, z$; (vii) $-x+1, -y, -z+1$; (viii) $x, y+1, z$; (ix) $x+1, y+1, z$; (x) $-x+2, -y, -z+2$; (xi) $-x, -y, -z+1$.