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1,4-Dibromo-2,5-di-*p*-toluoylbenzene

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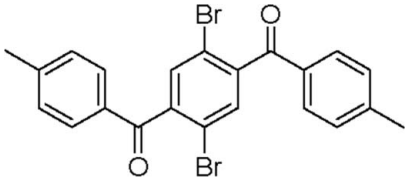
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.011 Å; R factor = 0.065; wR factor = 0.133; data-to-parameter ratio = 14.7.

In the title compound, $C_{22}H_{16}Br_2O_2$, which has approximate non-crystallographic inversion symmetry, the dihedral angles between the central ring and the pendant rings are 89.1 (4) and 82.4 (3)°; the dihedral angle between the pendant rings is 12.1 (4)°. In the crystal, the packing is influenced by van der Waals forces and no aromatic π–π stacking is observed.

Related literature

For background to the applications of the title compound, see: Shimizu *et al.* (2011). For further synthetic details, see: Chardonnens & Salamin (1968).



Experimental

Crystal data

$C_{22}H_{16}Br_2O_2$ M_r = 472.17
Monoclinic, $P2_1/n$
a = 9.855 (2) Å
b = 12.064 (2) Å
c = 16.345 (3) Å
β = 97.61 (3)°
V = 1926.2 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.22 mm^{-1}
T = 293 K
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.486, T_{max} = 0.678
3664 measured reflections
3452 independent reflections
1518 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
3 standard reflections every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.133
S = 1.00
3452 reflections
235 parameters
48 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors thank the Center of Test and Analysis, Nanjing University, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7121).

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supporting information

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1,4-Dibromo-2,5-di-*p*-toluoylbenzene

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S1. Comment

The title compound, 1,4-dibromo-2,5-di-*p*-toluoylbenzene is an important intermediate not only for manufacturing OLED materials, but also for sensing and switching devices that utilize solid-state luminescence as an output. (Shimizu *et al.*, 2011). We now report here its the crystal structure.

The molecular structure of (I) is shown in Fig. 1, and the selected geometric parameters are give in Table 1. The dihedral angles between the phenyl rings in *p*-toluoyl and the dibromobenzene are 89.14 (2)° and 82.41 (2)°. The phenyl rings are almost parallel with the maximum deviation of 0.26 (8)°. The crystal packing of the molecules in the crystal is influenced by van der Waals forces.

S2. Experimental

The title compund was synthesized according to the published procedure (Chardonnens & Salamin, 1968). Colourless blocks were obtained by dissolving it (0.5 g) in tetrahydrofuran (20 ml) and evaporating the solvent slowly at room temperature for about 10 d.

S3. Refinement

H atoms were positioned geometrically and refined as riding groups, with C—H = 0.93 Å for aromatic H, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C), where x = 1.2 for aromatic H, and x = 1.5 for other H.

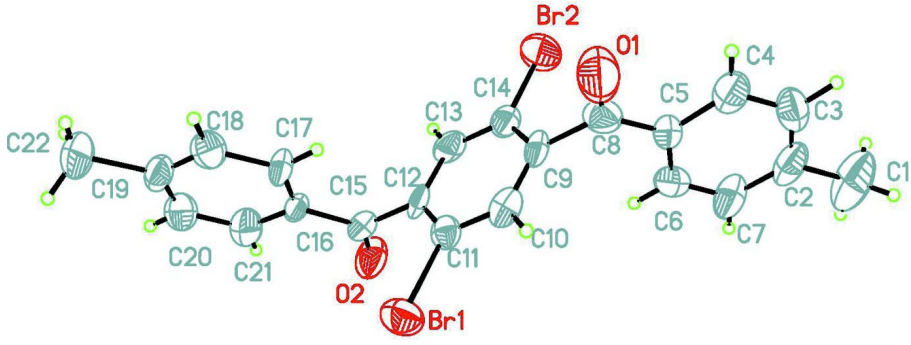

Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

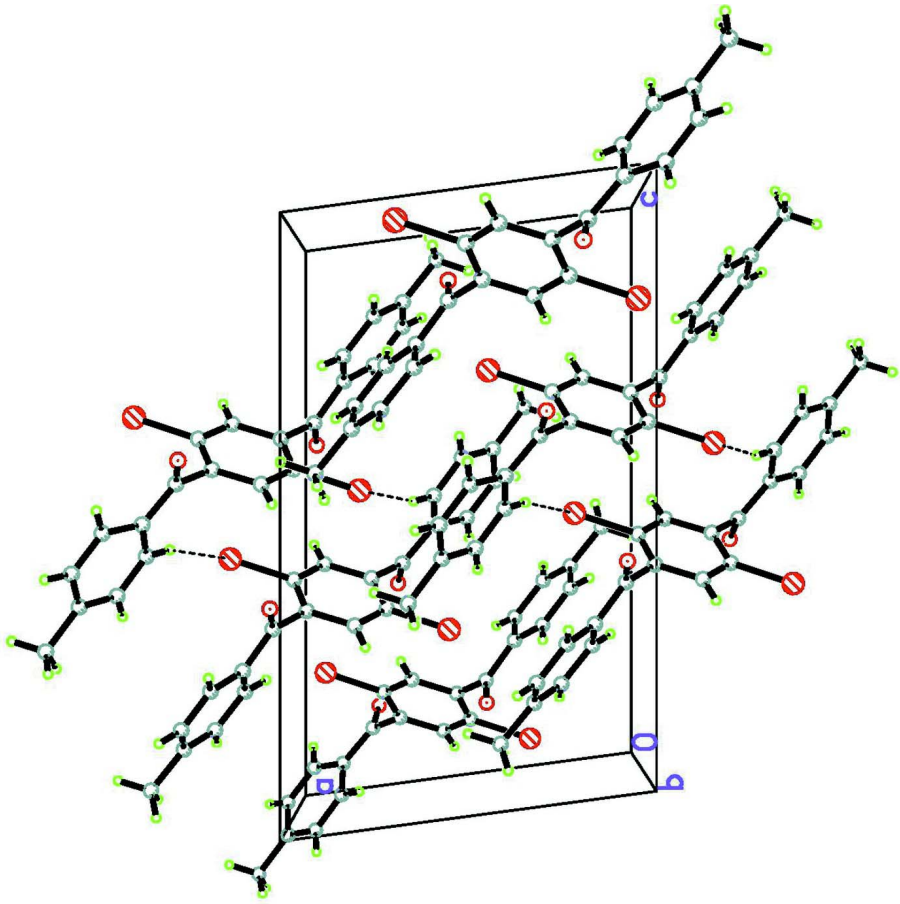

Figure 2

A packing diagram of (I).

1,4-Dibromo-2,5-di-*p*-toluoylbenzene

Crystal data

$C_{22}H_{16}Br_2O_2$
$M_r = 472.17$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.855$ (2) Å
$b = 12.064$ (2) Å
$c = 16.345$ (3) Å
$\beta = 97.61$ (3)°
$V = 1926.2$ (7) Å^3
$Z = 4$

$F(000) = 936$
$D_x = 1.628$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–13°
$\mu = 4.22$ mm^{-1}
$T = 293$ K
Block, colourless
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)

$T_{min} = 0.486$, $T_{max} = 0.678$
3664 measured reflections
3452 independent reflections
1518 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 25.2°$, $\theta_{min} = 2.1°$
$h = -11 \rightarrow 11$

$k = 0 \rightarrow 14$
$l = 0 \rightarrow 19$

3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.133$
$S = 1.00$
3452 reflections
235 parameters
48 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.050P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness 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 threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *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_{iso}*/U_{eq}
Br1	0.19197 (8)	0.40011 (8)	0.45593 (5)	0.0639 (3)
O1	−0.2881 (7)	0.1341 (5)	0.3699 (4)	0.095 (2)
C1	−0.6659 (9)	0.2445 (8)	0.6544 (5)	0.105 (4)
H1A	−0.6484	0.3109	0.6864	0.158*
H1B	−0.6483	0.1810	0.6897	0.158*
H1C	−0.7597	0.2436	0.6295	0.158*
Br2	−0.44399 (8)	0.39910 (9)	0.28265 (5)	0.0788 (4)
O2	0.0549 (6)	0.6656 (5)	0.3651 (3)	0.0740 (18)
C2	−0.5731 (9)	0.2412 (9)	0.5877 (5)	0.066 (3)
C3	−0.5798 (8)	0.1529 (7)	0.5314 (5)	0.060 (2)
H3A	−0.6427	0.0962	0.5349	0.072*
C4	−0.4954 (8)	0.1491 (7)	0.4718 (5)	0.059 (2)
H4A	−0.5023	0.0903	0.4347	0.071*
C5	−0.3983 (7)	0.2326 (7)	0.4658 (4)	0.045 (2)
C6	−0.3985 (8)	0.3206 (7)	0.5191 (5)	0.058 (2)
H6A	−0.3403	0.3800	0.5138	0.070*
C7	−0.4805 (10)	0.3237 (7)	0.5792 (5)	0.068 (3)
H7A	−0.4738	0.3832	0.6156	0.081*
C8	−0.3040 (8)	0.2209 (7)	0.4062 (5)	0.053 (2)
C9	−0.2155 (7)	0.3172 (6)	0.3889 (4)	0.0425 (18)
C10	−0.0778 (8)	0.3180 (6)	0.4244 (4)	0.0504 (19)
H10A	−0.0436	0.2619	0.4603	0.061*

C11	0.0067 (6)	0.4036 (6)	0.4052 (4)	0.0404 (17)
C12	−0.0358 (7)	0.4850 (6)	0.3498 (4)	0.0422 (18)
C13	−0.1739 (7)	0.4818 (6)	0.3136 (4)	0.0483 (19)
H13A	−0.2061	0.5361	0.2755	0.058*
C14	−0.2627 (7)	0.4003 (7)	0.3332 (4)	0.0464 (18)
C15	0.0537 (8)	0.5788 (7)	0.3286 (4)	0.051 (2)
C16	0.1406 (7)	0.5582 (7)	0.2634 (4)	0.0385 (18)
C17	0.1434 (7)	0.4579 (7)	0.2242 (4)	0.051 (2)
H17A	0.0876	0.4002	0.2374	0.062*
C18	0.2315 (7)	0.4427 (7)	0.1637 (4)	0.050 (2)
H18A	0.2341	0.3739	0.1383	0.060*
C19	0.3118 (7)	0.5248 (8)	0.1418 (4)	0.053 (2)
C20	0.3079 (8)	0.6253 (8)	0.1798 (5)	0.067 (3)
H20A	0.3628	0.6828	0.1652	0.080*
C21	0.2236 (8)	0.6427 (7)	0.2394 (5)	0.065 (2)
H21A	0.2220	0.7120	0.2642	0.078*
C22	0.4080 (8)	0.5080 (8)	0.0781 (4)	0.085 (3)
H22A	0.3981	0.4339	0.0567	0.127*
H22B	0.5006	0.5192	0.1033	0.127*
H22C	0.3864	0.5600	0.0339	0.127*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0470 (5)	0.0762 (6)	0.0651 (6)	−0.0002 (5)	−0.0061 (4)	0.0053 (5)
O1	0.116 (6)	0.083 (5)	0.098 (5)	−0.033 (4)	0.055 (4)	−0.022 (4)
C1	0.081 (7)	0.159 (11)	0.087 (7)	0.057 (7)	0.054 (6)	0.042 (7)
Br2	0.0463 (6)	0.1113 (8)	0.0740 (7)	−0.0026 (6)	−0.0098 (4)	0.0075 (6)
O2	0.091 (5)	0.074 (5)	0.064 (4)	−0.010 (4)	0.039 (3)	−0.018 (3)
C2	0.058 (6)	0.095 (8)	0.046 (5)	0.018 (5)	0.013 (5)	0.029 (6)
C3	0.045 (5)	0.065 (6)	0.072 (6)	−0.006 (4)	0.013 (5)	0.018 (5)
C4	0.054 (5)	0.076 (6)	0.049 (5)	0.005 (5)	0.010 (4)	0.011 (5)
C5	0.042 (5)	0.056 (5)	0.040 (5)	−0.009 (4)	0.012 (4)	0.002 (4)
C6	0.052 (5)	0.074 (7)	0.047 (5)	−0.013 (5)	0.002 (4)	−0.009 (5)
C7	0.094 (7)	0.062 (6)	0.053 (6)	0.004 (6)	0.033 (5)	0.006 (5)
C8	0.045 (5)	0.061 (6)	0.052 (5)	−0.003 (5)	0.008 (4)	−0.018 (5)
C9	0.046 (4)	0.051 (5)	0.035 (4)	−0.001 (4)	0.023 (3)	0.002 (3)
C10	0.063 (4)	0.051 (4)	0.040 (4)	0.005 (4)	0.015 (3)	0.008 (4)
C11	0.038 (4)	0.045 (4)	0.041 (4)	0.005 (3)	0.012 (3)	−0.003 (4)
C12	0.050 (4)	0.056 (5)	0.026 (4)	0.002 (4)	0.022 (3)	0.006 (3)
C13	0.054 (4)	0.058 (5)	0.032 (4)	0.010 (4)	0.000 (3)	0.014 (4)
C14	0.047 (4)	0.056 (4)	0.037 (4)	0.001 (4)	0.010 (3)	−0.001 (4)
C15	0.052 (5)	0.071 (7)	0.032 (5)	−0.005 (5)	0.011 (4)	−0.006 (4)
C16	0.034 (4)	0.058 (5)	0.025 (4)	0.005 (4)	0.008 (3)	0.001 (4)
C17	0.043 (5)	0.078 (6)	0.037 (5)	−0.001 (4)	0.023 (4)	−0.001 (4)
C18	0.046 (5)	0.050 (5)	0.055 (5)	0.005 (4)	0.007 (4)	0.001 (4)
C19	0.042 (5)	0.085 (7)	0.032 (5)	0.015 (5)	0.010 (4)	0.010 (5)
C20	0.063 (6)	0.083 (8)	0.056 (6)	−0.025 (5)	0.016 (5)	0.010 (5)

C21	0.065 (6)	0.074 (6)	0.060 (6)	−0.011 (5)	0.019 (5)	0.003 (5)
C22	0.070 (6)	0.136 (9)	0.054 (6)	−0.015 (6)	0.032 (5)	0.004 (6)

Geometric parameters (Å, º)

Br1—C11	1.904 (6)	C10—C11	1.388 (9)
O1—C8	1.224 (8)	C10—H10A	0.9300
C1—C2	1.514 (10)	C11—C12	1.363 (9)
C1—H1A	0.9600	C12—C13	1.410 (9)
C1—H1B	0.9600	C12—C15	1.503 (9)
C1—H1C	0.9600	C13—C14	1.382 (9)
Br2—C14	1.867 (7)	C13—H13A	0.9300
O2—C15	1.204 (8)	C15—C16	1.475 (9)
C2—C7	1.369 (11)	C16—C17	1.371 (9)
C2—C3	1.403 (11)	C16—C21	1.395 (10)
C3—C4	1.363 (9)	C17—C18	1.411 (9)
C3—H3A	0.9300	C17—H17A	0.9300
C4—C5	1.402 (10)	C18—C19	1.346 (9)
C4—H4A	0.9300	C18—H18A	0.9300
C5—C6	1.374 (9)	C19—C20	1.365 (10)
C5—C8	1.439 (9)	C19—C22	1.512 (9)
C6—C7	1.352 (9)	C20—C21	1.378 (10)
C6—H6A	0.9300	C20—H20A	0.9300
C7—H7A	0.9300	C21—H21A	0.9300
C8—C9	1.502 (10)	C22—H22A	0.9600
C9—C14	1.391 (9)	C22—H22B	0.9600
C9—C10	1.404 (9)	C22—H22C	0.9600
C2—C1—H1A	109.5	C11—C12—C13	117.0 (7)
C2—C1—H1B	109.5	C11—C12—C15	123.8 (7)
H1A—C1—H1B	109.5	C13—C12—C15	119.1 (7)
C2—C1—H1C	109.5	C14—C13—C12	121.8 (7)
H1A—C1—H1C	109.5	C14—C13—H13A	119.1
H1B—C1—H1C	109.5	C12—C13—H13A	119.1
C7—C2—C3	117.3 (8)	C13—C14—C9	119.8 (6)
C7—C2—C1	121.9 (9)	C13—C14—Br2	120.1 (6)
C3—C2—C1	120.8 (9)	C9—C14—Br2	120.1 (6)
C4—C3—C2	120.9 (8)	O2—C15—C16	122.5 (7)
C4—C3—H3A	119.5	O2—C15—C12	120.5 (7)
C2—C3—H3A	119.5	C16—C15—C12	117.0 (7)
C3—C4—C5	120.9 (8)	C17—C16—C21	117.7 (7)
C3—C4—H4A	119.6	C17—C16—C15	122.4 (7)
C5—C4—H4A	119.6	C21—C16—C15	119.9 (7)
C6—C5—C4	116.9 (7)	C16—C17—C18	119.6 (7)
C6—C5—C8	124.1 (8)	C16—C17—H17A	120.2
C4—C5—C8	119.0 (8)	C18—C17—H17A	120.2
C7—C6—C5	122.2 (8)	C19—C18—C17	121.9 (8)
C7—C6—H6A	118.9	C19—C18—H18A	119.0

C5—C6—H6A	118.9	C17—C18—H18A	119.0
C6—C7—C2	121.6 (9)	C18—C19—C20	118.7 (7)
C6—C7—H7A	119.2	C18—C19—C22	121.9 (8)
C2—C7—H7A	119.2	C20—C19—C22	119.4 (9)
O1—C8—C5	123.2 (8)	C19—C20—C21	120.8 (8)
O1—C8—C9	117.1 (7)	C19—C20—H20A	119.6
C5—C8—C9	119.7 (7)	C21—C20—H20A	119.6
C14—C9—C10	119.1 (7)	C20—C21—C16	121.2 (8)
C14—C9—C8	121.9 (7)	C20—C21—H21A	119.4
C10—C9—C8	118.8 (7)	C16—C21—H21A	119.4
C11—C10—C9	119.2 (7)	C19—C22—H22A	109.5
C11—C10—H10A	120.4	C19—C22—H22B	109.5
C9—C10—H10A	120.4	H22A—C22—H22B	109.5
C12—C11—C10	123.1 (7)	C19—C22—H22C	109.5
C12—C11—Br1	119.9 (5)	H22A—C22—H22C	109.5
C10—C11—Br1	117.0 (6)	H22B—C22—H22C	109.5

C7—C2—C3—C4	1.1 (12)	C11—C12—C13—C14	0.1 (10)
C1—C2—C3—C4	−179.3 (7)	C15—C12—C13—C14	−177.3 (7)
C2—C3—C4—C5	0.8 (12)	C12—C13—C14—C9	−1.3 (11)
C3—C4—C5—C6	−3.8 (11)	C12—C13—C14—Br2	−179.4 (5)
C3—C4—C5—C8	175.1 (7)	C10—C9—C14—C13	0.1 (10)
C4—C5—C6—C7	5.0 (11)	C8—C9—C14—C13	−174.2 (7)
C8—C5—C6—C7	−173.8 (7)	C10—C9—C14—Br2	178.2 (5)
C5—C6—C7—C2	−3.2 (13)	C8—C9—C14—Br2	3.9 (9)
C3—C2—C7—C6	0.0 (12)	C11—C12—C15—O2	−92.6 (10)
C1—C2—C7—C6	−179.6 (8)	C13—C12—C15—O2	84.6 (9)
C6—C5—C8—O1	166.2 (8)	C11—C12—C15—C16	86.1 (8)
C4—C5—C8—O1	−12.7 (12)	C13—C12—C15—C16	−96.7 (8)
C6—C5—C8—C9	−11.4 (11)	O2—C15—C16—C17	177.2 (7)
C4—C5—C8—C9	169.8 (7)	C12—C15—C16—C17	−1.4 (10)
O1—C8—C9—C14	98.5 (9)	O2—C15—C16—C21	−2.9 (11)
C5—C8—C9—C14	−83.8 (9)	C12—C15—C16—C21	178.4 (7)
O1—C8—C9—C10	−75.8 (9)	C21—C16—C17—C18	1.8 (10)
C5—C8—C9—C10	101.9 (8)	C15—C16—C17—C18	−178.4 (6)
C14—C9—C10—C11	2.1 (10)	C16—C17—C18—C19	−1.3 (11)
C8—C9—C10—C11	176.6 (6)	C17—C18—C19—C20	0.4 (11)
C9—C10—C11—C12	−3.4 (10)	C17—C18—C19—C22	178.9 (7)
C9—C10—C11—Br1	179.7 (5)	C18—C19—C20—C21	0.0 (12)
C10—C11—C12—C13	2.2 (10)	C22—C19—C20—C21	−178.5 (7)
Br1—C11—C12—C13	179.1 (5)	C19—C20—C21—C16	0.5 (12)
C10—C11—C12—C15	179.5 (7)	C17—C16—C21—C20	−1.4 (11)
Br1—C11—C12—C15	−3.6 (9)	C15—C16—C21—C20	178.7 (7)