data reports

CrossMark

2. Experimental

2.1. Crystal data

$C_{22}H_{28}Br_2O_2$	$V = 1092.91\ (12)$ Å^3
$M_r = 484.24$	$Z = 2$
Orthorhombic, $Pba2$	Mo $K\alpha$ radiation
$a = 7.3680\ (5)$ Å	$\mu = 3.72$ mm^{-1}
$b = 22.4243\ (14)$ Å	$T = 299$ K
$c = 6.6148\ (4)$ Å	$0.16 \times 0.15 \times 0.10$ mm

2.2. Data collection

Bruker D8 VENTURE diffractometer	9406 measured reflections
	1966 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)	1615 reflections with $I > 2\sigma(I)$
$T_{min} = 0.630$, $T_{max} = 0.773$	$R_{int} = 0.036$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$	$\Delta\rho_{min} = -0.21$ e Å^{-3}
$wR(F^2) = 0.065$	Absolute structure: Flack x
$S = 1.06$	determined using 621 quotients
1966 reflections	$[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons
123 parameters	*et al.*, 2013)
1 restraint	Absolute structure parameter:
H-atom parameters constrained	0.034 (9)
$\Delta\rho_{max} = 0.48$ e Å^{-3}	

Crystal structure of 5,5′-dibromo-3,3′-di-*tert*-butyl-6,6′-dimethylbiphenyl-2,2′-diol

Rika Obata,[a] Shigeru Ohba,[a]* Yasuaki Einaga[b] and Shigeru Nishiyama[b]

[a]Research and Education Center for Natural Sciences, Keio University, Hiyoshi 4-1-1, Kohoku-ku, Yokohama 223-8521, Japan, and [b]Department of Chemistry, Faculty of Science and Technology, Keio University; and JST-CREST/ACELL, Hiyoshi 3-14-1, Kohoku-ku, Yokohama 223-8522, Japan. *Correspondence e-mail: ohba@a3.keio.jp

The whole molecule of the title compound, $C_{22}H_{28}Br_2O_2$, is generated by twofold rotation symmetry. The dihedral angle of the biphenyl moiety is 85.05 (11)°. The hydroxy groups show intramolecular O—H···π interactions without any other hydrogen-bond acceptors. In the crystal, there are no other significant intermolecular interactions present.

1. Related literature

For the synthesis of the title compound using a transition-metal catalyst, see: Kubota *et al.* (2012). For the determination of the absolute configuration of the corresponding (+)-chloro derivative, *viz. S*, see: Gutierrez *et al.* (2010). For the crystal structure of a similar compound, *i.e.* 5,5′-dimethoxy-6,6′-dimethylbiphenyl-2,2′-diol dichloromethane solvate, see: Guo *et al.* (2011).

Table 1
Hydrogen-bond geometry (Å, °).

Cg is the centroid of benzene ring C3–C8.

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···Cg^i	0.82	2.54	3.047 (5)	122

Symmetry code: (i) $-x + 2, -y + 2, z$.

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL2014* and *publCIF* (Westrip, 2010).

Acknowledgements

Supporting information for this paper is available from the IUCr electronic archives (Reference: SU5104).

References

Bruker (2014). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Guo, F., Konkol, L. C. & Thomson, R. J. (2011). *J. Am. Chem. Soc.* **133**, 18–20.

Gutierrez, E. G., Moorhead, E. J., Smith, E. H., Lin, V., Ackerman, L. K. G., Knezevic, C. E., Sun, V., Grant, S. & Wenzel, A. G. (2010). *Eur. J. Org. Chem.* pp. 3027–3031.

Kubota, Y., Shirakawa, S., Inoue, T. & Maruoka, K. (2012). *Tetrahedron Lett.* **53**, 3739–3741.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Crystal structure of 5,5′-dibromo-3,3′-di-*tert*-butyl-6,6′-dimethylbiphenyl-2,2′-diol

Rika Obata, Shigeru Ohba, Yasuaki Einaga and Shigeru Nishiyama

S1. Synthesis and crystallization

The synthesis of the title compound, (I), is described in Fig. 2. It was prepared using iodine-mediated coupling method from 4-bromo-2-*tert*-butyl-5-methylphenol. To the solution of 4-bromo-2-*tert*-butyl-5-methylphenol (0.242 g, 1 mmol) in dichloromethane (1 mL) was added *N*-iodosuccinimide (abbreviated to NIS, 0.225 g, 1 mmol) and 3% H_2O_2 (1 mL). After shaking (200 rpm) the reaction mixture for 24 h at room temperature, it was poured into saturated $Na_2S_2O_3$ solution, and extracted with chloroform. The organic layer was washed with saturated NaCl and dried over anhydrous Na_2SO_4. The mixture was evaporated and purified by silica-gel column chromatography to give title compound (I) as white solid (yield: 0.138 g, 57%). ¹H-NMR (400 MHz, CDCl₃) 1.32 (18H, s), 1.92 (6H, s), 4.80 (2H, s), 7.47 (2H, s). Tof-MS ES(-) Anal. 481.0357, Calcd. 481.0378 for $C_{22}H_{27}O_2Br_2$. The crystals were grown by slow evaporation from a toluene/*n*-hexane (1/4) solution.

S2. Refinement

Crystal data, data collection and structure refinement details are summarized in the experimental table. The hydroxyl H atom was located from a difference Fourier map but was refined as riding (AFIX 147) with $U_{iso}(H) = 1.5U_{eq}(O)$. C-Bound H atoms were included in calculated positions and refined as riding: C—H = 0.93–0.96 Å with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C)$ for other H atoms.
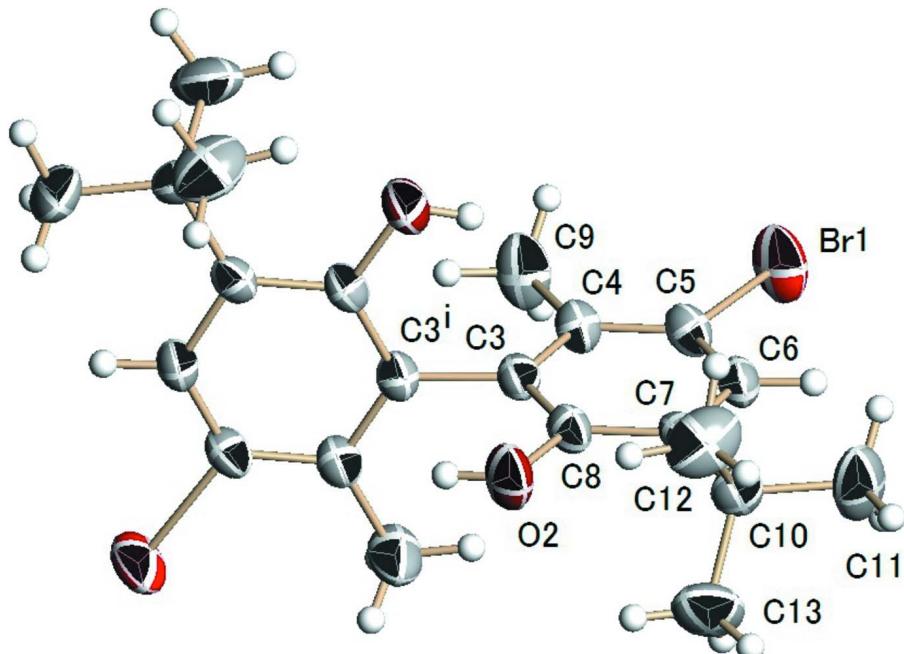
Figure 1

The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.
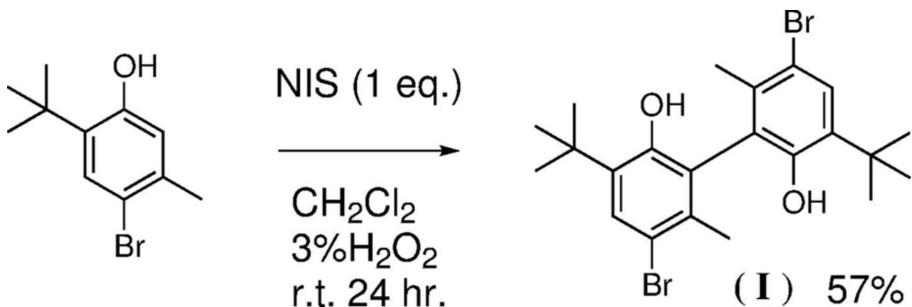
Figure 2

The synthesis of the title compound, (I).

5,5′-Dibromo-3,3′-di-*tert*-butyl-6,6′-dimethylbiphenyl-2,2′-diol

Crystal data

$C_{22}H_{28}Br_2O_2$	$D_x = 1.472$ Mg m^{-3}
$M_r = 484.24$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, $Pba2$	Cell parameters from 4121 reflections
$a = 7.3680 (5)$ Å	$\theta = 2.9–23.6°$
$b = 22.4243 (14)$ Å	$\mu = 3.72$ mm^{-1}
$c = 6.6148 (4)$ Å	$T = 299$ K
$V = 1092.91 (12)$ Å^3	Prism, colourless
$Z = 2$	$0.16 \times 0.15 \times 0.10$ mm
$F(000) = 492$	

Data collection

Bruker D8 VENTURE diffractometer	1966 independent reflections
Radiation source: fine-focus sealed tube	1615 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.036$
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)	$\theta_{max} = 25.3°$, $\theta_{min} = 2.9°$
	$h = -8\rightarrow8$
	$k = -26\rightarrow26$
$T_{min} = 0.630$, $T_{max} = 0.773$	$l = -7\rightarrow7$
9406 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.065$	$w = 1/[\sigma^2(F_o^2) + (0.0176P)^2 + 0.3778P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	
1966 reflections	$(\Delta/\sigma)_{max} = 0.001$
123 parameters	$\Delta\rho_{max} = 0.48$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack x determined using 621 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
	Absolute structure parameter: 0.034 (9)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	1.18668 (8)	0.81211 (2)	0.48288 (15)	0.0681 (2)
O2	0.7528 (4)	0.98204 (13)	0.9897 (9)	0.0576 (8)
H2	0.7777	1.0166	0.9607	0.086*
C3	0.9856 (6)	0.96676 (17)	0.7429 (7)	0.0352 (10)
C4	1.0903 (6)	0.9290 (2)	0.6223 (7)	0.0388 (11)
C5	1.0536 (6)	0.8689 (2)	0.6397 (7)	0.0399 (11)
C6	0.9212 (6)	0.84644 (19)	0.7649 (7)	0.0394 (11)
H6	0.9023	0.8054	0.7685	0.047*
C7	0.8150 (6)	0.88292 (19)	0.8859 (7)	0.0360 (10)
C8	0.8518 (6)	0.94415 (19)	0.8707 (7)	0.0370 (11)
C9	1.2322 (7)	0.9538 (2)	0.4852 (15)	0.0650 (14)
H9A	1.1993	0.9460	0.3473	0.098*
H9B	1.3467	0.9353	0.5143	0.098*
H9C	1.2417	0.9960	0.5058	0.098*
C10	0.6668 (5)	0.8582 (2)	1.0234 (8)	0.0428 (15)
C11	0.6564 (7)	0.7905 (2)	1.0111 (14)	0.074 (2)
H11A	0.6243	0.7789	0.8761	0.111*
H11B	0.5661	0.7762	1.1039	0.111*
H11C	0.7722	0.7737	1.0456	0.111*

C12	0.7053 (8)	0.8746 (3)	1.2449 (9)	0.0756 (18)
H12A	0.8260	0.8623	1.2802	0.113*
H12B	0.6195	0.8548	1.3311	0.113*
H12C	0.6944	0.9170	1.2619	0.113*
C13	0.4827 (6)	0.8829 (2)	0.9625 (15)	0.0760 (17)
H13A	0.4791	0.9249	0.9894	0.114*
H13B	0.3893	0.8632	1.0387	0.114*
H13C	0.4634	0.8760	0.8208	0.114*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0866 (4)	0.0458 (3)	0.0718 (4)	0.0118 (2)	0.0311 (4)	−0.0118 (4)
O2	0.0646 (19)	0.0366 (16)	0.072 (2)	0.0049 (14)	0.018 (3)	−0.009 (3)
C3	0.040 (3)	0.030 (2)	0.036 (3)	0.0016 (19)	−0.004 (2)	0.001 (2)
C4	0.048 (3)	0.033 (3)	0.035 (3)	0.003 (2)	0.002 (2)	0.000 (2)
C5	0.048 (3)	0.035 (3)	0.037 (3)	0.009 (2)	0.003 (2)	−0.003 (2)
C6	0.046 (3)	0.028 (2)	0.045 (3)	0.002 (2)	−0.002 (2)	−0.002 (2)
C7	0.036 (3)	0.034 (2)	0.038 (2)	0.008 (2)	−0.005 (2)	−0.002 (2)
C8	0.042 (3)	0.031 (3)	0.038 (2)	0.006 (2)	0.000 (2)	−0.005 (2)
C9	0.084 (3)	0.048 (3)	0.063 (3)	0.003 (2)	0.033 (5)	−0.003 (5)
C10	0.035 (3)	0.045 (3)	0.048 (4)	−0.0010 (19)	0.008 (3)	0.003 (2)
C11	0.070 (3)	0.048 (3)	0.104 (6)	−0.013 (2)	0.036 (5)	0.008 (4)
C12	0.078 (4)	0.099 (5)	0.049 (4)	−0.021 (4)	0.010 (3)	0.002 (3)
C13	0.039 (3)	0.086 (4)	0.103 (5)	0.005 (2)	0.012 (4)	0.020 (5)

Geometric parameters (Å, º)

Br1—C5	1.913 (4)	C9—H9B	0.9600
O2—C8	1.369 (6)	C9—H9C	0.9600
O2—H2	0.8200	C10—C13	1.520 (7)
C3—C8	1.394 (6)	C10—C11	1.521 (7)
C3—C4	1.396 (6)	C10—C12	1.537 (8)
C3—C3ⁱ	1.506 (8)	C11—H11A	0.9600
C4—C5	1.379 (6)	C11—H11B	0.9600
C4—C9	1.491 (8)	C11—H11C	0.9600
C5—C6	1.375 (6)	C12—H12A	0.9600
C6—C7	1.386 (6)	C12—H12B	0.9600
C6—H6	0.9300	C12—H12C	0.9600
C7—C8	1.403 (6)	C13—H13A	0.9600
C7—C10	1.526 (6)	C13—H13B	0.9600
C9—H9A	0.9600	C13—H13C	0.9600
C8—O2—H2	109.5	C13—C10—C11	107.7 (4)
C8—C3—C4	121.1 (4)	C13—C10—C7	110.4 (5)
C8—C3—C3ⁱ	117.3 (4)	C11—C10—C7	111.5 (4)
C4—C3—C3ⁱ	121.6 (4)	C13—C10—C12	109.3 (5)
C5—C4—C3	115.9 (4)	C11—C10—C12	107.4 (5)

C5—C4—C9	123.5 (4)	C7—C10—C12	110.5 (4)
C3—C4—C9	120.5 (4)	C10—C11—H11A	109.5
C6—C5—C4	123.2 (4)	C10—C11—H11B	109.5
C6—C5—Br1	116.5 (3)	H11A—C11—H11B	109.5
C4—C5—Br1	120.3 (3)	C10—C11—H11C	109.5
C5—C6—C7	122.2 (4)	H11A—C11—H11C	109.5
C5—C6—H6	118.9	H11B—C11—H11C	109.5
C7—C6—H6	118.9	C10—C12—H12A	109.5
C6—C7—C8	115.3 (4)	C10—C12—H12B	109.5
C6—C7—C10	122.2 (4)	H12A—C12—H12B	109.5
C8—C7—C10	122.5 (4)	C10—C12—H12C	109.5
O2—C8—C3	120.0 (4)	H12A—C12—H12C	109.5
O2—C8—C7	117.6 (4)	H12B—C12—H12C	109.5
C3—C8—C7	122.4 (4)	C10—C13—H13A	109.5
C4—C9—H9A	109.5	C10—C13—H13B	109.5
C4—C9—H9B	109.5	H13A—C13—H13B	109.5
H9A—C9—H9B	109.5	C10—C13—H13C	109.5
C4—C9—H9C	109.5	H13A—C13—H13C	109.5
H9A—C9—H9C	109.5	H13B—C13—H13C	109.5
H9B—C9—H9C	109.5		
C8—C3—C4—C5	−0.5 (6)	C3^i—C3—C8—O2	0.0 (6)
C3^i—C3—C4—C5	178.5 (4)	C4—C3—C8—C7	0.1 (7)
C8—C3—C4—C9	179.6 (5)	C3^i—C3—C8—C7	−179.0 (4)
C3^i—C3—C4—C9	−1.4 (7)	C6—C7—C8—O2	−178.9 (4)
C3—C4—C5—C6	0.9 (7)	C10—C7—C8—O2	1.9 (6)
C9—C4—C5—C6	−179.3 (6)	C6—C7—C8—C3	0.1 (6)
C3—C4—C5—Br1	179.9 (3)	C10—C7—C8—C3	−179.1 (4)
C9—C4—C5—Br1	−0.2 (7)	C6—C7—C10—C13	−118.6 (5)
C4—C5—C6—C7	−0.8 (7)	C8—C7—C10—C13	60.5 (6)
Br1—C5—C6—C7	−179.8 (3)	C6—C7—C10—C11	1.0 (6)
C5—C6—C7—C8	0.3 (7)	C8—C7—C10—C11	−179.8 (5)
C5—C6—C7—C10	179.4 (4)	C6—C7—C10—C12	120.4 (5)
C4—C3—C8—O2	179.0 (4)	C8—C7—C10—C12	−60.5 (6)

Symmetry code: (i) −*x*+2, −*y*+2, *z*.

Hydrogen-bond geometry (Å, º)

Cg is the centroid of benzene ring C3–C8.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···*Cg*^{i}	0.82	2.54	3.047 (5)	122

Symmetry code: (i) −*x*+2, −*y*+2, *z*.