Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,3′-Dibromo-5,5′-di-*tert*-butyl-2,2′-dimethoxybiphenyl

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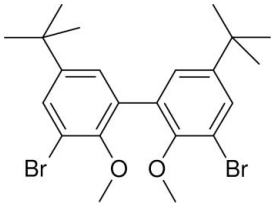
Received 9 November 2007; accepted 22 January 2008

Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.010 Å; disorder in main residue; R factor = 0.067; wR factor = 0.143; data-to-parameter ratio = 13.4.

The title compound, $C_{22}H_{28}Br_2O_2$, crystallizes in a staggered arrangement to minimize the interactions of its *ortho* substituents, with a dihedral angle of 84.2 (3)° between the two aromatic rings. Short C—H···O hydrogen-bonding interactions between methoxy groups result in a one-dimensional polymeric chain of molecules lying parallel to the *b* axis. One *tert*-butyl group is disordered equally over two positions.

Related literature

For a related structure, see: He & Ng (2006); Steiner (1996). For an alternative synthesis, see: Katagiri *et al.* (2006).



Experimental

Crystal data

$C_{22}H_{28}Br_2O_2$
M_r = 484.27
Monoclinic, $C2/c$
a = 14.661 (2) Å
b = 13.408 (2) Å
c = 22.489 (3) Å
β = 96.104 (12)°
V = 4395.8 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.70 mm^{-1}
T = 93 (2) K
0.40 × 0.12 × 0.10 mm

Data collection

Bruker APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.433, T_{max} = 0.691
14546 measured reflections
3918 independent reflections
2784 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.143
S = 1.10
3918 reflections
293 parameters
54 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.93 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C31—H31C···O10^{i}	0.98	2.61	2.842 (15)	94
C41—H41C···O21^{ii}	0.98	2.57	2.866 (14)	98

Symmetry codes: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Version 1.08; Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2008).

We thank the Foundation of Research Science and Technology for funding. PJS also thanks the Royal Society of New Zealand for the award of a James Cook Research Fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2045).

References

Bruker (2007). *APEX2* (Version 2.1-4), *SAINT* (Version 7.34A), *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
He, L. & Ng, S. W. (2006). *Acta Cryst.* E**62**, o5517–o5519.
Katagiri, H., Miyagawa, T., Furusho, Y. & Yashima, E. (2006). *Angew. Chem. Int. Ed.* **45**, 1741–1744.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Steiner, T. (1996). *Cryst. Rev.* **6**, 1–57.
Westrip, S. P. (2008). *publCIF.* In preparation.

  doi:10.1107/S1600536808002420

supporting information

Acta Cryst. (2008). E**64**, o532 [doi:10.1107/S1600536808002420]

3,3′-Dibromo-5,5′-di-*tert*-butyl-2,2′-dimethoxybiphenyl

Matthew I. J. Polson and Peter J. Steel

S1. Comment

During attempts to dilithiate 2,6-dibromo-4 - t-butylanisole, the title compound, (I), was serindipitously produced. Compared to the literature methods (Katagiri *et al.*, 2006) this is a much simpler method, wherein the product was acheived in a single step rather than three and with a superior overall yield. The structure adopts a staggered arrangement with a dihedral angle of 84.2 (3)° between the two aromatic rings. A similar angle is found in the literature (80.1°, He & Ng, 2006). Two CH_3···O hydrogen bonds involving both methoxy groups (Table 1) connect the molecules to form a one dimensional polymeric chain parallel to the *b* axis (Figure 2); similar type of interactions have already been reported (Steiner, 1996).

S2. Experimental

2,6-Dibromo-4 - t-butylanisole (1 g) in THF (40 ml) at 193 K was treated with *n*-butyl lithium (1.6 *M*, 2.5 ml). The solution was stirred and allowed to warm up to room temp over 2 hr. The resulting solution was evaporated to dryness, treated with water and extracted with dichloromethane. The organic layer was seperated and purified by column chromotography (SiO_2, dichloromethane). Yeild = 0.6 g (79%).

S3. Refinement

The methoxy groups are both evenly disordered over two sites. One *tert*-butyl group is disordered over two sites whilst the other is not. This breaks the potential symmetry between the two halves of the molecule. Both *tert*-butyl groups exhibited elongation of the thermal elipsoids and have been restrained (ISOR) to be more isotropic. The large redidual electron density (1.11 e/A*3) is located 0.64Å from H70A and is probably related to a small amount of unmodelled *tert*-butyl group disorder. All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.95 Å, $U_{iso} = 1.2U_{eq}$(C) for aromatic, and 0.98 Å, $U_{iso} = 1.5U_{eq}$(C) for CH_3 atoms.

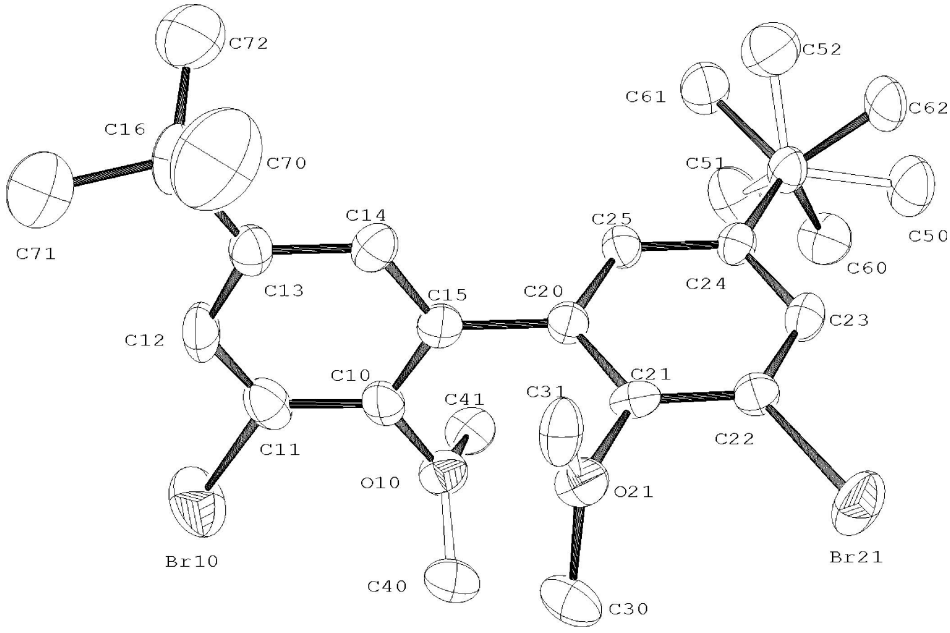


Figure 1

A view of the asymmetric unit of (I), showing displacement ellipsoids at the 50% probability level. All hydrogen atoms have been omited for clarity. The bonds for one of the disordered parts are displayed as hollow bonds.

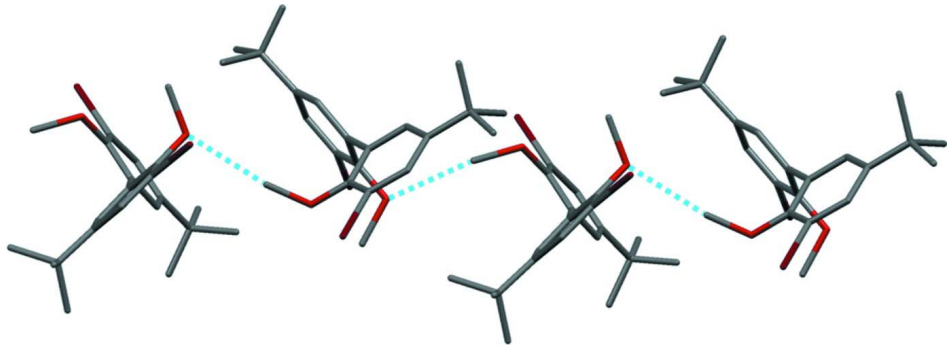


Figure 2

A diagram showing the hydrogen bonding which extends the structure into a 1-D polymer. The closest C—H···O bonds are shown as dashed lines.

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Crystal data

$C_{22}H_{28}Br_2O_2$
$M_r = 484.27$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 14.661\ (2)$ Å
$b = 13.408\ (2)$ Å
$c = 22.489\ (3)$ Å
$\beta = 96.104\ (12)°$
$V = 4395.8\ (11)$ Å^3
$Z = 8$
$F(000) = 1968$
$D_x = 1.463$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4881 reflections
$\theta = 2.8–26.5°$
$\mu = 3.70$ mm^{-1}
$T = 93$ K
Shard, colourless
0.40 × 0.12 × 0.10 mm

Data collection

Bruker APEX2 CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.433, T_{max} = 0.691
14546 measured reflections
3918 independent reflections
2784 reflections with $I > 2\sigma(I)$
R_{int} = 0.077
θ_{max} = 25.1°, θ_{min} = 2.1°
h = −17→17
k = −14→16
l = −18→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.143
S = 1.10
3918 reflections
293 parameters
54 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
$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.5007P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.93 e Å^{-3}

Special details

Experimental. Spectroscopic data: ^{1}H NMR ($CDCl_3$): δ 1.33 (18*H*, s, $(CH_3)_3$), 3.53 (6*H*, s, OCH_3), 7.36 (2*H*, d, ArH), 7.57 (2*H*, d, ArH). Mass Spec: (ESI-TOF) 485.3 {*M*+} calc 485.05.

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}	Occ. (<1)
Br10	0.15107 (7)	0.73599 (8)	0.05847 (4)	0.0641 (3)	
C10	0.2414 (5)	0.7782 (5)	0.1739 (3)	0.0318 (16)	
C11	0.2365 (5)	0.8029 (6)	0.1143 (3)	0.0384 (18)	
C12	0.2911 (5)	0.8769 (6)	0.0943 (3)	0.0395 (18)	
H12	0.2846	0.8937	0.0531	0.047*	
C13	0.3553 (5)	0.9273 (5)	0.1330 (3)	0.0319 (16)	
C14	0.3623 (5)	0.8991 (5)	0.1932 (3)	0.0305 (16)	
H14	0.4073	0.9302	0.2206	0.037*	
C15	0.3053 (4)	0.8267 (5)	0.2143 (3)	0.0278 (15)	
C16	0.4181 (6)	1.0090 (5)	0.1119 (3)	0.0404 (18)	
Br21	0.18307 (6)	0.87188 (6)	0.42817 (3)	0.0464 (2)	
C20	0.3129 (4)	0.7995 (5)	0.2790 (3)	0.0264 (15)	
C21	0.2538 (4)	0.8409 (5)	0.3166 (3)	0.0264 (15)	
C22	0.2626 (4)	0.8137 (5)	0.3765 (3)	0.0268 (15)	

C23	0.3269 (4)	0.7445 (5)	0.3990 (3)	0.0285 (15)	
H23	0.3308	0.7263	0.4400	0.034*	
C24	0.3863 (4)	0.7008 (5)	0.3618 (3)	0.0247 (14)	
C25	0.3790 (4)	0.7306 (5)	0.3025 (3)	0.0265 (14)	
H25	0.4202	0.7035	0.2769	0.032*	
C26	0.4557 (5)	0.6205 (5)	0.3860 (3)	0.0317 (16)	
O21	0.1887 (3)	0.9095 (3)	0.2945 (2)	0.0332 (11)	
C30	0.0955 (9)	0.8615 (11)	0.2798 (7)	0.042 (4)	0.50
H30A	0.1009	0.8049	0.2528	0.062*	0.50
H30B	0.0526	0.9105	0.2603	0.062*	0.50
H30C	0.0726	0.8379	0.3167	0.062*	0.50
C31	0.2269 (10)	1.0158 (10)	0.2987 (6)	0.037 (3)	0.50
H31A	0.2469	1.0319	0.3405	0.055*	0.50
H31B	0.1790	1.0628	0.2830	0.055*	0.50
H31C	0.2792	1.0208	0.2751	0.055*	0.50
O10	0.1857 (3)	0.7052 (3)	0.1937 (2)	0.0349 (11)	
C40	0.0985 (9)	0.7443 (12)	0.2011 (7)	0.043 (4)	0.50
H40A	0.1045	0.7958	0.2322	0.064*	0.50
H40B	0.0587	0.6908	0.2130	0.064*	0.50
H40C	0.0716	0.7736	0.1634	0.064*	0.50
C41	0.2209 (10)	0.5993 (9)	0.1949 (6)	0.036 (3)	0.50
H41A	0.2306	0.5786	0.1542	0.054*	0.50
H41B	0.1760	0.5550	0.2105	0.054*	0.50
H41C	0.2791	0.5955	0.2206	0.054*	0.50
C50	0.4475 (12)	0.5886 (13)	0.4517 (7)	0.050 (4)	0.50
H50A	0.4877	0.5314	0.4619	0.075*	0.50
H50B	0.3838	0.5699	0.4559	0.075*	0.50
H50C	0.4657	0.6442	0.4786	0.075*	0.50
C51	0.4383 (12)	0.5241 (12)	0.3484 (7)	0.052 (4)	0.50
H51A	0.4576	0.5346	0.3085	0.078*	0.50
H51B	0.3728	0.5078	0.3449	0.078*	0.50
H51C	0.4735	0.4690	0.3682	0.078*	0.50
C52	0.5535 (11)	0.6565 (13)	0.3834 (8)	0.053 (4)	0.50
H52A	0.5635	0.7177	0.4070	0.080*	0.50
H52B	0.5636	0.6699	0.3418	0.080*	0.50
H52C	0.5965	0.6050	0.3999	0.080*	0.50
C60	0.4038 (11)	0.5287 (11)	0.3985 (7)	0.043 (4)	0.50
H60A	0.3881	0.4918	0.3612	0.065*	0.50
H60B	0.3474	0.5473	0.4155	0.065*	0.50
H60C	0.4416	0.4866	0.4269	0.065*	0.50
C61	0.5257 (11)	0.6015 (12)	0.3413 (7)	0.045 (4)	0.50
H61A	0.5465	0.6654	0.3264	0.067*	0.50
H61B	0.4971	0.5619	0.3077	0.067*	0.50
H61C	0.5784	0.5651	0.3612	0.067*	0.50
C62	0.5094 (10)	0.6633 (11)	0.4442 (6)	0.038 (3)	0.50
H62A	0.5527	0.6131	0.4618	0.057*	0.50
H62B	0.4661	0.6806	0.4728	0.057*	0.50
H62C	0.5431	0.7232	0.4343	0.057*	0.50

C70	0.4117 (9)	1.0992 (9)	0.1512 (5)	0.103 (4)
H70A	0.4498	1.0889	0.1892	0.155*
H70B	0.3477	1.1092	0.1588	0.155*
H70C	0.4333	1.1582	0.1311	0.155*
C71	0.3892 (7)	1.0442 (8)	0.0495 (4)	0.079 (3)
H71A	0.4268	1.1015	0.0404	0.119*
H71B	0.3245	1.0640	0.0461	0.119*
H71C	0.3973	0.9902	0.0212	0.119*
C72	0.5157 (6)	0.9717 (7)	0.1155 (4)	0.065 (3)
H72A	0.5559	1.0256	0.1047	0.098*
H72B	0.5193	0.9157	0.0879	0.098*
H72C	0.5353	0.9495	0.1564	0.098*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br10	0.0766 (7)	0.0720 (7)	0.0396 (5)	−0.0096 (5)	−0.0125 (4)	−0.0105 (5)
C10	0.030 (4)	0.038 (4)	0.027 (4)	0.015 (3)	0.003 (3)	0.001 (3)
C11	0.040 (4)	0.046 (5)	0.029 (4)	0.013 (4)	−0.002 (3)	−0.007 (3)
C12	0.051 (5)	0.049 (5)	0.019 (3)	0.008 (4)	0.006 (3)	0.004 (3)
C13	0.035 (4)	0.034 (4)	0.027 (4)	0.008 (3)	0.005 (3)	0.003 (3)
C14	0.033 (4)	0.028 (4)	0.032 (4)	0.002 (3)	0.009 (3)	0.001 (3)
C15	0.028 (4)	0.026 (4)	0.030 (4)	0.010 (3)	0.007 (3)	0.000 (3)
C16	0.065 (5)	0.029 (4)	0.029 (4)	0.004 (4)	0.012 (4)	0.004 (3)
Br21	0.0562 (5)	0.0481 (5)	0.0387 (4)	0.0201 (4)	0.0232 (3)	0.0043 (4)
C20	0.026 (4)	0.028 (4)	0.025 (3)	−0.003 (3)	0.002 (3)	0.000 (3)
C21	0.020 (3)	0.021 (3)	0.039 (4)	0.001 (3)	0.007 (3)	0.003 (3)
C22	0.026 (4)	0.026 (4)	0.029 (4)	0.002 (3)	0.004 (3)	−0.001 (3)
C23	0.035 (4)	0.028 (4)	0.023 (3)	−0.005 (3)	0.007 (3)	−0.003 (3)
C24	0.028 (4)	0.022 (3)	0.024 (3)	−0.002 (3)	0.005 (3)	0.001 (3)
C25	0.028 (4)	0.028 (4)	0.023 (3)	−0.001 (3)	0.001 (3)	0.000 (3)
C26	0.035 (4)	0.033 (4)	0.028 (4)	0.008 (3)	0.006 (3)	0.008 (3)
O21	0.032 (3)	0.027 (3)	0.041 (3)	0.004 (2)	0.008 (2)	0.007 (2)
C30	0.035 (8)	0.034 (9)	0.054 (9)	0.008 (7)	−0.009 (7)	0.008 (7)
C31	0.057 (10)	0.033 (8)	0.022 (7)	0.006 (7)	0.009 (7)	−0.003 (6)
O10	0.033 (3)	0.034 (3)	0.038 (3)	0.002 (2)	0.006 (2)	0.000 (2)
C40	0.027 (8)	0.053 (10)	0.046 (9)	−0.008 (7)	−0.004 (6)	0.000 (8)
C41	0.039 (8)	0.022 (8)	0.047 (9)	−0.013 (6)	0.004 (7)	0.005 (6)
C50	0.059 (8)	0.052 (8)	0.039 (7)	0.015 (7)	0.008 (6)	0.018 (6)
C51	0.062 (8)	0.040 (7)	0.050 (7)	0.015 (7)	−0.006 (7)	−0.001 (6)
C52	0.045 (7)	0.056 (8)	0.059 (8)	0.007 (7)	0.008 (6)	0.015 (7)
C60	0.047 (7)	0.031 (7)	0.052 (7)	−0.004 (6)	0.004 (6)	0.008 (6)
C61	0.042 (7)	0.053 (8)	0.039 (7)	0.015 (6)	0.007 (6)	0.000 (6)
C62	0.046 (7)	0.038 (7)	0.029 (6)	0.006 (6)	0.001 (6)	0.014 (6)
C70	0.126 (6)	0.085 (5)	0.104 (5)	−0.007 (4)	0.035 (4)	0.003 (4)
C71	0.075 (5)	0.090 (5)	0.072 (5)	−0.009 (4)	0.010 (4)	0.027 (4)
C72	0.059 (4)	0.058 (4)	0.078 (4)	−0.011 (4)	0.005 (4)	0.019 (4)

Geometric parameters (Å, º)

Br10—C11	1.902 (7)	C31—H31B	0.9800
C10—C11	1.375 (9)	C31—H31C	0.9800
C10—O10	1.379 (8)	O10—C40	1.408 (15)
C10—C15	1.394 (9)	O10—C41	1.511 (14)
C11—C12	1.379 (10)	C40—H40A	0.9800
C12—C13	1.388 (10)	C40—H40B	0.9800
C12—H12	0.9500	C40—H40C	0.9800
C13—C14	1.398 (9)	C41—H41A	0.9800
C13—C16	1.539 (10)	C41—H41B	0.9800
C14—C15	1.397 (9)	C41—H41C	0.9800
C14—H14	0.9500	C50—H50A	0.9800
C15—C20	1.493 (9)	C50—H50B	0.9800
C16—C71	1.499 (11)	C50—H50C	0.9800
C16—C70	1.507 (13)	C51—H51A	0.9800
C16—C72	1.510 (11)	C51—H51B	0.9800
Br21—C22	1.899 (6)	C51—H51C	0.9800
C20—C21	1.390 (9)	C52—H52A	0.9800
C20—C25	1.401 (9)	C52—H52B	0.9800
C21—O21	1.380 (7)	C52—H52C	0.9800
C21—C22	1.388 (9)	C60—H60A	0.9800
C22—C23	1.380 (9)	C60—H60B	0.9800
C23—C24	1.398 (9)	C60—H60C	0.9800
C23—H23	0.9500	C61—H61A	0.9800
C24—C25	1.385 (8)	C61—H61B	0.9800
C24—C26	1.541 (9)	C61—H61C	0.9800
C25—H25	0.9500	C62—H62A	0.9800
C26—C60	1.489 (15)	C62—H62B	0.9800
C26—C52	1.519 (17)	C62—H62C	0.9800
C26—C61	1.533 (15)	C70—H70A	0.9800
C26—C51	1.550 (17)	C70—H70B	0.9800
C26—C50	1.555 (15)	C70—H70C	0.9800
C26—C62	1.563 (15)	C71—H71A	0.9800
O21—C30	1.516 (14)	C71—H71B	0.9800
O21—C31	1.530 (15)	C71—H71C	0.9800
C30—H30A	0.9800	C72—H72A	0.9800
C30—H30B	0.9800	C72—H72B	0.9800
C30—H30C	0.9800	C72—H72C	0.9800
C31—H31A	0.9800		

C11—C10—O10	121.0 (6)	O21—C30—H30A	109.5
C11—C10—C15	119.0 (7)	O21—C30—H30B	109.5
O10—C10—C15	120.0 (6)	O21—C30—H30C	109.5
C10—C11—C12	121.3 (7)	O21—C31—H31A	109.5
C10—C11—Br10	119.5 (6)	O21—C31—H31B	109.5
C12—C11—Br10	119.2 (5)	H31A—C31—H31B	109.5
C11—C12—C13	121.6 (6)	O21—C31—H31C	109.5

C11—C12—H12	119.2	H31A—C31—H31C	109.5
C13—C12—H12	119.2	H31B—C31—H31C	109.5
C12—C13—C14	116.8 (6)	C10—O10—C40	110.5 (8)
C12—C13—C16	122.8 (6)	C10—O10—C41	117.3 (7)
C14—C13—C16	120.5 (6)	C40—O10—C41	131.4 (9)
C15—C14—C13	122.1 (6)	O10—C40—H40A	109.5
C15—C14—H14	118.9	O10—C40—H40B	109.5
C13—C14—H14	118.9	H40A—C40—H40B	109.5
C10—C15—C14	119.2 (6)	O10—C40—H40C	109.5
C10—C15—C20	119.8 (6)	H40A—C40—H40C	109.5
C14—C15—C20	121.1 (6)	H40B—C40—H40C	109.5
C71—C16—C70	105.5 (8)	O10—C41—H41A	109.5
C71—C16—C72	109.2 (7)	O10—C41—H41B	109.5
C70—C16—C72	110.6 (8)	O10—C41—H41C	109.5
C71—C16—C13	113.3 (7)	C26—C50—H50A	109.5
C70—C16—C13	108.3 (7)	C26—C50—H50B	109.5
C72—C16—C13	110.0 (6)	C26—C50—H50C	109.5
C21—C20—C25	119.0 (6)	C26—C51—H51A	109.5
C21—C20—C15	120.6 (6)	C26—C51—H51B	109.5
C25—C20—C15	120.4 (6)	C26—C51—H51C	109.5
O21—C21—C22	121.0 (5)	C26—C52—H52A	109.5
O21—C21—C20	120.0 (6)	C26—C52—H52B	109.5
C22—C21—C20	119.0 (6)	C26—C52—H52C	109.5
C23—C22—C21	121.5 (6)	C26—C60—H60A	109.5
C23—C22—Br21	119.5 (5)	C26—C60—H60B	109.5
C21—C22—Br21	119.0 (5)	H60A—C60—H60B	109.5
C22—C23—C24	120.4 (6)	C26—C60—H60C	109.5
C22—C23—H23	119.8	H60A—C60—H60C	109.5
C24—C23—H23	119.8	H60B—C60—H60C	109.5
C25—C24—C23	117.8 (6)	C26—C61—H61A	109.5
C25—C24—C26	121.5 (5)	C26—C61—H61B	109.5
C23—C24—C26	120.7 (5)	H61A—C61—H61B	109.5
C24—C25—C20	122.2 (6)	C26—C61—H61C	109.5
C24—C25—H25	118.9	H61A—C61—H61C	109.5
C20—C25—H25	118.9	H61B—C61—H61C	109.5
C60—C26—C52	140.6 (10)	C26—C62—H62A	109.5
C60—C26—C61	112.2 (10)	C26—C62—H62B	109.5
C52—C26—C61	47.7 (9)	H62A—C62—H62B	109.5
C60—C26—C24	108.2 (8)	C26—C62—H62C	109.5
C52—C26—C24	110.8 (8)	H62A—C62—H62C	109.5
C61—C26—C24	110.4 (7)	H62B—C62—H62C	109.5
C60—C26—C51	49.9 (9)	C16—C70—H70A	109.5
C52—C26—C51	110.2 (11)	C16—C70—H70B	109.5
C61—C26—C51	65.6 (10)	H70A—C70—H70B	109.5
C24—C26—C51	109.0 (8)	C16—C70—H70C	109.5
C60—C26—C50	60.0 (9)	H70A—C70—H70C	109.5
C52—C26—C50	107.0 (10)	H70B—C70—H70C	109.5
C61—C26—C50	134.6 (9)	C16—C71—H71A	109.5

C24—C26—C50	114.4 (7)	C16—C71—H71B	109.5
C51—C26—C50	105.3 (10)	H71A—C71—H71B	109.5
C60—C26—C62	111.6 (9)	C16—C71—H71C	109.5
C52—C26—C62	61.4 (9)	H71A—C71—H71C	109.5
C61—C26—C62	107.3 (9)	H71B—C71—H71C	109.5
C24—C26—C62	107.1 (7)	C16—C72—H72A	109.5
C51—C26—C62	143.3 (9)	C16—C72—H72B	109.5
C50—C26—C62	52.4 (8)	H72A—C72—H72B	109.5
C21—O21—C30	111.6 (7)	C16—C72—H72C	109.5
C21—O21—C31	111.4 (7)	H72A—C72—H72C	109.5
C30—O21—C31	136.5 (8)	H72B—C72—H72C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C31—H31*C*···O10^{i}	0.98	2.61	2.842 (15)	94
C41—H41*C*···O21^{ii}	0.98	2.57	2.866 (14)	98

Symmetry codes: (i) −*x*+1/2, *y*+1/2, −*z*+1/2; (ii) −*x*+1/2, *y*−1/2, −*z*+1/2.