Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

9-(4-Bromophenyl)-3,3,6,6-tetramethyl-3,4,6,7-tetrahydro-2*H*-xanthene-1,8(5*H*,9*H*)-dione

M. Ali Bigdeli,[a]* Gholam Hossein Mahdavinia[a] and Vahid Amani[b]

[a]Department of Chemistry, Teacher Training University, 49 Mofateh Avenue, 15614 Tehran, Iran, and [b]Academy of Scientific Studies in Education, 16 Hojjat Dost Street, Vessal Shirazi Avenue, Tehran, Iran
Correspondence e-mail: mabig397@yahoo.com

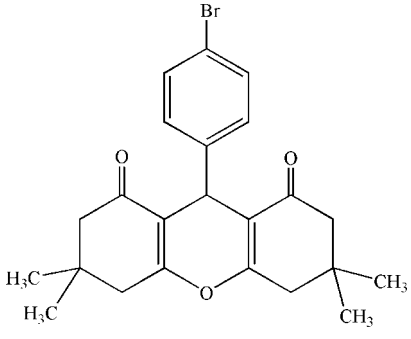
Received 18 June 2007; accepted 9 July 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.077; data-to-parameter ratio = 19.4.

The title compound, $C_{23}H_{25}BrO_3$, was synthesized by the reaction of *p*-bromobenzaldehyde with dimedone and $HClO_4$/SiO_2 in EtOH. In the molecule, the dihydropyran ring adopts a boat conformation and the two cyclohexene rings are in a *trans* conformation.

Related literature

For related crystal structures, see: Tu *et al.* (2002, 2004); Jeyakanthan *et al.* (1999); Li *et al.* (2005); Shi *et al.* (1997). For related literature, see: Casiraghi *et al.* (1973); Hideo (1981); Ion *et al.* (2000); Knight & Little (2001); Lambert *et al.* (1997); Menchen *et al.* (2003); Poupelin *et al.* (1978); Wang & Harvey (2002).



Experimental

Crystal data

$C_{23}H_{25}BrO_3$
M_r = 429.34
Monoclinic, $P2_1/n$
a = 5.9667 (6) Å
b = 19.5626 (18) Å
c = 17.389 (2) Å
β = 97.488 (5)°
V = 2012.5 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.06 mm^{-1}
T = 100 (2) K
0.30 × 0.25 × 0.25 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADADS*; Sheldrick, 2003)
T_{min} = 0.541, T_{max} = 0.596
10510 measured reflections
4809 independent reflections
3885 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.077
S = 1.00
4809 reflections
248 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the Teacher Training University and the Academy of Scientific Studies in Education for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2439).

References

Bruker (2001). *SAINT-Plus*. Version 6.2. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Casiraghi, G., Casnati, G. & Cornia, M. (1973). *Tetrahedron Lett.* **14**, 679–682.
Hideo, T. (1981). Jpn Patent JP 56 005 480.
Ion, R. M., Albulescu, C., Sirkecioglu, O. & Talinli, N. (2000). *Internet Photochemistry and Photobiology*. URL: http://www.photobiology.com/photobiology2000/rodica2/index.htm
Jeyakanthan, J., Shanmuga Sundara Raj, S., Velmurugan, D., Fun, H.-K. & Murugan, P. (1999). *Acta Cryst.* C**55**, 1515–1517.
Knight, D. W. & Little, P. B. (2001). *J. Chem. Soc. Perkin Trans. 1*, pp. 1771–1777.
Lambert, R. W., Martin, J. A., Merrett, J. H., Parkes, K. E. B. & Thomas, G. J. (1997). PCT Int. Appl. WO 9 706 178.
Li, T. J., Tu, S. J., Zhu, S. L. & Zhang, X. J. (2005). *Jiegou Huaxue*, **24**, 25–29.
Menchen, S. M., Benson, S. C., Lam, J. Y. L., Zhen, W., Sun, D., Rosenblum, B. B., Khan, S. H. & Taing, M. (2003). US Patent 6 583 168.
Poupelin, J. P., Saint, R. G., Foussard, B. O., Narcisse, G., Uchida, E. G. & Lacroix, R. (1978). *Eur. J. Med. Chem.* **13**, 67–71.
Sheldrick, G. M. (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2003). *SADABS*. Version 2.03. Bruker AXS Inc, Madison, Wisconsin, USA.
Shi, D. Q., Mu, L. L., Lu, Z. S., Gao, Y. G. & Dai, G. Y. (1997). *Jiegou Huaxue*, **16**, 484–487.
Tu, S. J., Miao, C. B., Fang, F. & Jiang, H. (2004). *Jiegou Huaxue*, **23**, 187–191.
Tu, S., Zhou, J., Lu, Z., Deng, X., Shi, D. & Wang, S. (2002). *Synth. Commun.* **32**, 3063–3067.
Wang, J. Q. & Harvey, R. G. (2002). *Tetrahedron*, **58**, 5927–5931.

supplementary materials

Acta Cryst. (2007). E**63**, o3493 [**doi:10.1107/S1600536807033545**]

9-(4-Bromophenyl)-3,3,6,6-tetramethyl-3,4,6,7-tetrahydro-2*H*-xanthene-1,8(5*H*,9*H*)-dione

M. A. Bigdeli, G. H. Mahdavinia and V. Amani

Comment

Xanthenes and benzoxanthenes are important classes of compounds that find uses as dyes and fluorescent materials for visualization of bio-molecules and laser technologies due to their useful spectroscopic properties (Menchen *et al.*, 2003). Xanthene-based compounds have also been investigated for agricultural bactericide activity (Hideo, 1981), photodynamic therapy (Ion *et al.*, 2000), anti-inflammatory effect (Poupelin *et al.*, 1987) and antiviral activity (Lambert *et al.*, 1997). Various literature procedures are available to synthesis xanthenes including palladium catalyzed cyclization of polycyclic aryltriflate esters (Wang & Harvey, 2002), intramolecular trapping of benzynes by phenols (Knight & Little, 2001) and reaction of aryloxymagnesium halides with triethylorthoformate (Casiraghi *et al.*, 2003). However, these methodologies suffer from one or more disadvantages such as low yield, lack of easy availability or preparation of the starting materials, prolonged reaction time (16 h to 5 days), use of toxic organic solvents, requirement of excess of reagents or catalysts, special apparatus and harsh reaction conditions. In the light of the above, we have synthesized the title compound.

The bond lengths and angles in the title molecule (Fig. 1) are comparable with those reported for related structures (Tu *et al.*, 2002; Jeyakanthan *et al.*, 1999; Li *et al.*, 2005; Shi *et al.*, 1997; Tu *et al.*, 2004). The pyran rings have a half-chair conformations; atoms C5 and C13 lie 0.471Å and 0.533Å from the mean plane through atoms C6/C7/C2/C3 and C14/C15/C10/C11 respectively, while atoms C4 and C12 are only 0.189Å and 0.158 Å from these planes. The dihedral angles between the benzene ring of the bromo-phenyl group the main planes of the pyran rings are 78.67 (16)° and 87.33 (18)° for C2—C7 and C10—C15 respectively.

Experimental

A mixture of *p*-Bromobenzaldehyde (1 mmol), dimedone (2 mmol) and $HClO_4/SiO_2$ (20 mg, 0.01 mmol, 1 mol%) was stirred in solvent free condition at 253 K for appropriate time (15 min). The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was dissolved in hot ethanol and the catalyst was separated by simple filtration. The filtrate was kept at room temperature to give the pure product. The milky precipitated product was recrystallized from EtOH. After one day, colorless prismatic crystals were isolated (yield 86%; m.p. 506–508 K).

Refinement

The H atom positions were calculated [C—H = 0.95–1.00 Å] and they were refined in an isotropic riding-model approximation with U_{iso}(H) = 1.2U_{eq}(C) or 1.5 U_{eq}(C) for methyl groups.

Figures

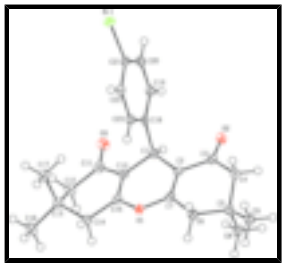


Fig. 1. The molecular structure. Displacement ellipsoids are drawn at the 50% probability level

9-(4-Bromophenyl)-3,3,6,6-tetramethyl-3,4,6,7-tetrahydro-2*H*-xanthene- 1,8(5*H*,9H)-dione

Crystal data

$C_{23}H_{25}BrO_3$	F_{000} = 888
M_r = 429.34	D_x = 1.417 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 475 reflections
a = 5.9667 (6) Å	θ = 3–30º
b = 19.5626 (18) Å	μ = 2.06 mm^{-1}
c = 17.389 (2) Å	T = 100 (2) K
β = 97.488 (5)º	Plate, colourless
V = 2012.5 (4) Å^3	0.30 × 0.25 × 0.25 mm
Z = 4	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	4809 independent reflections
Radiation source: fine-focus sealed tube	3885 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.028
T = 100(2) K	θ_{max} = 28.0º
φ and ω scans	θ_{min} = 1.6º
Absorption correction: multi-scan (SADADS; Sheldrick, 2003)	h = −7→7
T_{min} = 0.541, T_{max} = 0.596	k = −25→11
10510 measured reflections	l = −22→20

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.031	H-atom parameters constrained
$wR(F^2)$ = 0.077	$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.432P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.00	$(\Delta/\sigma)_{max}$ = 0.001
4809 reflections	$\Delta\rho_{max}$ = 0.46 e Å^{-3}
248 parameters	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	1.07429 (3)	0.101879 (10)	1.012508 (11)	0.02169 (7)
O1	1.0166 (2)	0.30464 (7)	0.62322 (8)	0.0153 (3)
O2	0.6944 (2)	0.40950 (7)	0.82823 (8)	0.0226 (3)
O3	0.3829 (2)	0.18458 (7)	0.69070 (8)	0.0225 (3)
C1	0.7060 (3)	0.28876 (9)	0.73700 (10)	0.0133 (4)
H1A	0.5487	0.3038	0.7425	0.016*
C2	0.8510 (3)	0.35134 (9)	0.72954 (11)	0.0141 (4)
C3	0.8399 (3)	0.40817 (10)	0.78432 (11)	0.0166 (4)
C4	1.0180 (3)	0.46320 (10)	0.78548 (11)	0.0187 (4)
H4A	1.1539	0.4486	0.8204	0.022*
H4B	0.9603	0.5054	0.8074	0.022*
C5	1.0877 (3)	0.47985 (10)	0.70564 (11)	0.0153 (4)
C6	1.1546 (3)	0.41280 (9)	0.66840 (12)	0.0172 (4)
H6A	1.1613	0.4208	0.6125	0.021*
H6B	1.3078	0.3994	0.6925	0.021*
C7	0.9954 (3)	0.35544 (9)	0.67685 (11)	0.0139 (4)
C8	0.8913 (3)	0.51453 (10)	0.65452 (12)	0.0208 (4)
H8A	0.8539	0.5577	0.6785	0.031*
H8B	0.7590	0.4844	0.6492	0.031*
H8C	0.9354	0.5237	0.6032	0.031*
C9	1.2920 (3)	0.52753 (10)	0.71582 (12)	0.0201 (4)
H9A	1.2500	0.5709	0.7381	0.030*
H9B	1.3425	0.5360	0.6653	0.030*
H9C	1.4148	0.5062	0.7506	0.030*
C10	0.7008 (3)	0.24655 (9)	0.66414 (10)	0.0132 (4)
C11	0.5247 (3)	0.19420 (10)	0.64695 (11)	0.0158 (4)
C12	0.5197 (3)	0.15530 (10)	0.57155 (11)	0.0176 (4)

H12A	0.4286	0.1814	0.5298	0.021*
H12B	0.4434	0.1109	0.5766	0.021*
C13	0.7542 (3)	0.14199 (10)	0.54758 (11)	0.0158 (4)
C14	0.8750 (3)	0.21109 (9)	0.54547 (11)	0.0148 (4)
H14A	1.0369	0.2030	0.5416	0.018*
H14B	0.8100	0.2364	0.4985	0.018*
C15	0.8556 (3)	0.25385 (9)	0.61514 (11)	0.0135 (4)
C16	0.7290 (4)	0.11028 (11)	0.46627 (12)	0.0247 (5)
H16A	0.6499	0.0664	0.4669	0.037*
H16B	0.8791	0.1029	0.4506	0.037*
H16C	0.6421	0.1412	0.4294	0.037*
C17	0.8883 (3)	0.09338 (10)	0.60568 (12)	0.0208 (4)
H17A	0.8089	0.0495	0.6058	0.031*
H17B	0.9025	0.1135	0.6577	0.031*
H17C	1.0391	0.0860	0.5906	0.031*
C18	0.7980 (3)	0.24673 (9)	0.80815 (11)	0.0132 (4)
C19	0.6719 (3)	0.23501 (10)	0.86853 (11)	0.0159 (4)
H19A	0.5281	0.2561	0.8675	0.019*
C20	0.7523 (3)	0.19291 (10)	0.93060 (11)	0.0171 (4)
H20A	0.6638	0.1844	0.9712	0.021*
C21	0.9646 (3)	0.16371 (9)	0.93175 (10)	0.0152 (4)
C22	1.0978 (3)	0.17662 (9)	0.87388 (11)	0.0150 (4)
H22A	1.2449	0.1575	0.8765	0.018*
C23	1.0130 (3)	0.21796 (10)	0.81204 (11)	0.0153 (4)
H23A	1.1026	0.2268	0.7718	0.018*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02593 (11)	0.02092 (11)	0.01725 (11)	0.00010 (8)	−0.00079 (8)	0.00554 (8)
O1	0.0155 (6)	0.0123 (6)	0.0191 (7)	−0.0014 (5)	0.0064 (5)	−0.0013 (5)
O2	0.0278 (8)	0.0190 (7)	0.0232 (8)	0.0021 (6)	0.0111 (6)	−0.0018 (6)
O3	0.0178 (7)	0.0264 (8)	0.0252 (8)	−0.0045 (6)	0.0094 (6)	−0.0022 (6)
C1	0.0123 (8)	0.0123 (9)	0.0164 (9)	0.0015 (7)	0.0057 (7)	−0.0002 (7)
C2	0.0144 (9)	0.0120 (9)	0.0158 (9)	0.0016 (7)	0.0011 (7)	0.0021 (7)
C3	0.0204 (9)	0.0141 (9)	0.0152 (9)	0.0048 (7)	0.0019 (8)	0.0026 (7)
C4	0.0249 (10)	0.0158 (9)	0.0155 (9)	−0.0004 (8)	0.0028 (8)	−0.0016 (7)
C5	0.0163 (9)	0.0122 (9)	0.0171 (9)	0.0001 (7)	0.0008 (7)	0.0008 (7)
C6	0.0179 (9)	0.0151 (9)	0.0196 (10)	0.0002 (7)	0.0057 (8)	0.0001 (7)
C7	0.0150 (9)	0.0118 (9)	0.0147 (9)	0.0033 (7)	0.0012 (7)	0.0008 (7)
C8	0.0233 (10)	0.0158 (9)	0.0226 (10)	0.0024 (8)	0.0007 (8)	0.0028 (8)
C9	0.0229 (10)	0.0144 (9)	0.0227 (10)	−0.0027 (8)	0.0020 (8)	−0.0004 (8)
C10	0.0129 (8)	0.0138 (9)	0.0129 (9)	0.0017 (7)	0.0015 (7)	0.0017 (7)
C11	0.0114 (8)	0.0176 (9)	0.0183 (10)	0.0021 (7)	0.0013 (7)	0.0012 (7)
C12	0.0126 (9)	0.0210 (10)	0.0190 (10)	−0.0027 (8)	0.0014 (7)	−0.0026 (8)
C13	0.0139 (9)	0.0166 (9)	0.0170 (9)	−0.0015 (7)	0.0028 (7)	−0.0009 (7)
C14	0.0165 (9)	0.0141 (9)	0.0143 (9)	0.0013 (7)	0.0035 (7)	0.0015 (7)
C15	0.0127 (8)	0.0115 (9)	0.0160 (9)	0.0006 (7)	0.0006 (7)	0.0025 (7)

C16	0.0257 (11)	0.0283 (12)	0.0205 (10)	−0.0050 (9)	0.0043 (8)	−0.0098 (9)
C17	0.0198 (10)	0.0173 (10)	0.0257 (11)	−0.0005 (8)	0.0042 (8)	0.0012 (8)
C18	0.0156 (9)	0.0100 (8)	0.0141 (9)	−0.0016 (7)	0.0028 (7)	−0.0027 (7)
C19	0.0150 (9)	0.0151 (9)	0.0183 (10)	0.0003 (7)	0.0046 (7)	−0.0008 (7)
C20	0.0184 (9)	0.0179 (9)	0.0164 (9)	−0.0020 (8)	0.0074 (8)	−0.0010 (7)
C21	0.0215 (9)	0.0107 (9)	0.0126 (9)	−0.0035 (7)	−0.0011 (7)	−0.0003 (7)
C22	0.0117 (8)	0.0137 (9)	0.0193 (9)	−0.0002 (7)	0.0004 (7)	−0.0028 (7)
C23	0.0159 (9)	0.0160 (9)	0.0150 (9)	−0.0012 (7)	0.0054 (7)	−0.0006 (7)

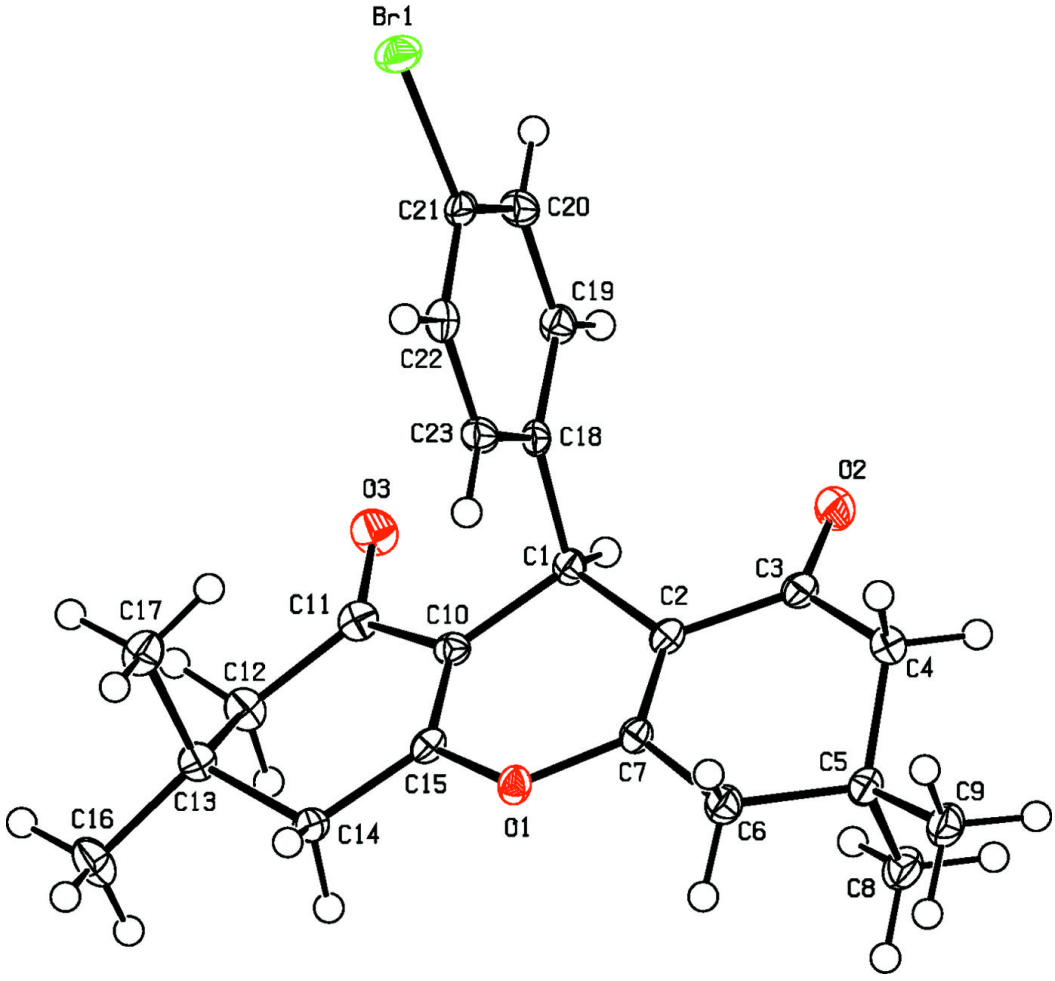
Geometric parameters (Å, °)

Br1—C21	1.9037 (18)	C10—C11	1.470 (3)
O1—C15	1.376 (2)	C11—C12	1.513 (3)
O1—C7	1.380 (2)	C12—C13	1.533 (3)
O2—C3	1.229 (2)	C12—H12A	0.9900
O3—C11	1.224 (2)	C12—H12B	0.9900
C1—C10	1.509 (3)	C13—C16	1.533 (3)
C1—C2	1.514 (3)	C13—C17	1.534 (3)
C1—C18	1.527 (3)	C13—C14	1.535 (3)
C1—H1A	1.0000	C14—C15	1.489 (3)
C2—C7	1.340 (3)	C14—H14A	0.9900
C2—C3	1.471 (3)	C14—H14B	0.9900
C3—C4	1.511 (3)	C16—H16A	0.9800
C4—C5	1.535 (3)	C16—H16B	0.9800
C4—H4A	0.9900	C16—H16C	0.9800
C4—H4B	0.9900	C17—H17A	0.9800
C5—C9	1.527 (3)	C17—H17B	0.9800
C5—C8	1.534 (3)	C17—H17C	0.9800
C5—C6	1.539 (3)	C18—C19	1.388 (3)
C6—C7	1.490 (3)	C18—C23	1.394 (3)
C6—H6A	0.9900	C19—C20	1.392 (3)
C6—H6B	0.9900	C19—H19A	0.9500
C8—H8A	0.9800	C20—C21	1.387 (3)
C8—H8B	0.9800	C20—H20A	0.9500
C8—H8C	0.9800	C21—C22	1.385 (3)
C9—H9A	0.9800	C22—C23	1.387 (3)
C9—H9B	0.9800	C22—H22A	0.9500
C9—H9C	0.9800	C23—H23A	0.9500
C10—C15	1.343 (3)		
C15—O1—C7	117.69 (14)	C11—C12—C13	113.92 (15)
C10—C1—C2	108.65 (15)	C11—C12—H12A	108.8
C10—C1—C18	110.57 (15)	C13—C12—H12A	108.8
C2—C1—C18	110.83 (14)	C11—C12—H12B	108.8
C10—C1—H1A	108.9	C13—C12—H12B	108.8
C2—C1—H1A	108.9	H12A—C12—H12B	107.7
C18—C1—H1A	108.9	C12—C13—C16	109.65 (15)
C7—C2—C3	118.99 (17)	C12—C13—C17	110.01 (16)
C7—C2—C1	122.19 (17)	C16—C13—C17	109.65 (16)
C3—C2—C1	118.76 (16)	C12—C13—C14	107.77 (16)

O2—C3—C2	120.88 (18)	C16—C13—C14	108.90 (16)
O2—C3—C4	121.95 (18)	C17—C13—C14	110.81 (15)
C2—C3—C4	117.15 (17)	C15—C14—C13	112.87 (16)
C3—C4—C5	114.33 (16)	C15—C14—H14A	109.0
C3—C4—H4A	108.7	C13—C14—H14A	109.0
C5—C4—H4A	108.7	C15—C14—H14B	109.0
C3—C4—H4B	108.7	C13—C14—H14B	109.0
C5—C4—H4B	108.7	H14A—C14—H14B	107.8
H4A—C4—H4B	107.6	C10—C15—O1	122.71 (17)
C9—C5—C8	109.59 (16)	C10—C15—C14	126.09 (17)
C9—C5—C4	109.33 (15)	O1—C15—C14	111.18 (15)
C8—C5—C4	109.97 (16)	C13—C16—H16A	109.5
C9—C5—C6	108.81 (15)	C13—C16—H16B	109.5
C8—C5—C6	110.71 (16)	H16A—C16—H16B	109.5
C4—C5—C6	108.40 (15)	C13—C16—H16C	109.5
C7—C6—C5	113.37 (16)	H16A—C16—H16C	109.5
C7—C6—H6A	108.9	H16B—C16—H16C	109.5
C5—C6—H6A	108.9	C13—C17—H17A	109.5
C7—C6—H6B	108.9	C13—C17—H17B	109.5
C5—C6—H6B	108.9	H17A—C17—H17B	109.5
H6A—C6—H6B	107.7	C13—C17—H17C	109.5
C2—C7—O1	122.87 (17)	H17A—C17—H17C	109.5
C2—C7—C6	126.02 (17)	H17B—C17—H17C	109.5
O1—C7—C6	111.10 (16)	C19—C18—C23	118.85 (17)
C5—C8—H8A	109.5	C19—C18—C1	121.92 (16)
C5—C8—H8B	109.5	C23—C18—C1	119.21 (16)
H8A—C8—H8B	109.5	C18—C19—C20	121.21 (18)
C5—C8—H8C	109.5	C18—C19—H19A	119.4
H8A—C8—H8C	109.5	C20—C19—H19A	119.4
H8B—C8—H8C	109.5	C21—C20—C19	118.45 (18)
C5—C9—H9A	109.5	C21—C20—H20A	120.8
C5—C9—H9B	109.5	C19—C20—H20A	120.8
H9A—C9—H9B	109.5	C22—C21—C20	121.61 (17)
C5—C9—H9C	109.5	C22—C21—Br1	118.50 (14)
H9A—C9—H9C	109.5	C20—C21—Br1	119.86 (14)
H9B—C9—H9C	109.5	C21—C22—C23	118.93 (17)
C15—C10—C11	118.52 (17)	C21—C22—H22A	120.5
C15—C10—C1	122.26 (17)	C23—C22—H22A	120.5
C11—C10—C1	119.19 (16)	C22—C23—C18	120.88 (17)
O3—C11—C10	120.96 (18)	C22—C23—H23A	119.6
O3—C11—C12	121.63 (17)	C18—C23—H23A	119.6
C10—C11—C12	117.35 (16)		
C10—C1—C2—C7	−16.9 (2)	C1—C10—C11—C12	177.34 (16)
C18—C1—C2—C7	104.8 (2)	O3—C11—C12—C13	−147.91 (18)
C10—C1—C2—C3	165.88 (15)	C10—C11—C12—C13	34.8 (2)
C18—C1—C2—C3	−72.4 (2)	C11—C12—C13—C16	−173.17 (17)
C7—C2—C3—O2	172.65 (17)	C11—C12—C13—C17	66.2 (2)
C1—C2—C3—O2	−10.1 (3)	C11—C12—C13—C14	−54.8 (2)
C7—C2—C3—C4	−9.0 (2)	C12—C13—C14—C15	46.4 (2)

C1—C2—C3—C4	168.34 (16)	C16—C13—C14—C15	165.24 (16)
O2—C3—C4—C5	−145.33 (18)	C17—C13—C14—C15	−74.1 (2)
C2—C3—C4—C5	36.3 (2)	C11—C10—C15—O1	175.09 (16)
C3—C4—C5—C9	−171.30 (16)	C1—C10—C15—O1	−6.7 (3)
C3—C4—C5—C8	68.3 (2)	C11—C10—C15—C14	−3.3 (3)
C3—C4—C5—C6	−52.8 (2)	C1—C10—C15—C14	174.92 (17)
C9—C5—C6—C7	162.90 (16)	C7—O1—C15—C10	−9.7 (2)
C8—C5—C6—C7	−76.6 (2)	C7—O1—C15—C14	168.91 (15)
C4—C5—C6—C7	44.1 (2)	C13—C14—C15—C10	−19.5 (3)
C3—C2—C7—O1	−179.88 (16)	C13—C14—C15—O1	161.93 (15)
C1—C2—C7—O1	2.9 (3)	C10—C1—C18—C19	−120.65 (18)
C3—C2—C7—C6	1.2 (3)	C2—C1—C18—C19	118.80 (19)
C1—C2—C7—C6	−175.97 (17)	C10—C1—C18—C23	58.0 (2)
C15—O1—C7—C2	11.6 (2)	C2—C1—C18—C23	−62.6 (2)
C15—O1—C7—C6	−169.34 (15)	C23—C18—C19—C20	−2.8 (3)
C5—C6—C7—C2	−20.5 (3)	C1—C18—C19—C20	175.76 (17)
C5—C6—C7—O1	160.52 (15)	C18—C19—C20—C21	1.3 (3)
C2—C1—C10—C15	18.7 (2)	C19—C20—C21—C22	1.3 (3)
C18—C1—C10—C15	−103.1 (2)	C19—C20—C21—Br1	−176.57 (14)
C2—C1—C10—C11	−163.02 (15)	C20—C21—C22—C23	−2.2 (3)
C18—C1—C10—C11	75.1 (2)	Br1—C21—C22—C23	175.70 (14)
C15—C10—C11—O3	178.39 (18)	C21—C22—C23—C18	0.6 (3)
C1—C10—C11—O3	0.1 (3)	C19—C18—C23—C22	1.9 (3)
C15—C10—C11—C12	−4.3 (2)	C1—C18—C23—C22	−176.73 (16)

Fig. 1


Br1
C21
C20
C19
C22
C23
C18
O3
O2
C1
C3
C17
C11
C10
C2
C4
C12
C5
C9
C15
C7
C13
C16
C14
O1
C6
C8