Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 5-bromo-9-ethoxy-2-oxo-2*H*-pyrano-[2,3-*g*]quinoline-8-carboxylate

César D. de Oliveira,[a] Gilberto A. Romeiro,[a] Janet M. S. Skakle,[b]* James L. Wardell[c] and Solange M. S. V. Wardell[d]

[a]Instituto de Química, Departamento de Química Inorgânica, Universidade Federal Fluminense, Outeiro de São João Batista s/n, Campus do Valonguinho–Centro, Niterói, RJ 24020-150, Brazil, [b]Department of Chemistry, College of Physical Sciences, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland, [c]Departamento de Química Inorgânica, Instituto de Química, Universidade Federal do Rio de Janeiro, 21945-970 Rio de Janeiro, RJ, Brazil, and [d]Laboratory of Organic Synthesis of Far-Manguinhos/FIOCRUZ, R. Sizenando Nabuco, 100 Manguinhos, 21041-250 Rio de Janeiro, RJ, Brazil

Correspondence e-mail: j.skakle@abdn.ac.uk

Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.004 Å
R factor = 0.032
wR factor = 0.069
Data-to-parameter ratio = 24.2

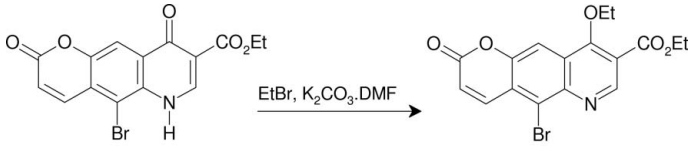
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 15 February 2006
Accepted 27 February 2006

The title compound, $C_{17}H_{14}BrNO_5$, was studied as part of a study on the biological properties of pyranoquinoline derivatives. Alkylation of the parent pyranoquinoline was shown to have occurred at the carbonyl rather than the amino site. The molecules are linked by a C—H···O hydrogen bond forming *C*(5) chains along [001].

Comment

As part of a study on the synthesis and biological activities of pyranoquinoline derivatives (da Matta *et al.*, 2000; de Oliveira, 2003), alkylation of compound (1) (see scheme) using EtBr was carried out. Compound (1) contains two potential reaction sites, *viz*. the carbonyl and amino sites. While NMR spectroscopy strongly indicated that bromination had occurred predominantly at the carbonyl site to give the pyranoquinoline derivative (2), confirmation was sought using X-ray crystallography.



(1) (2)

Structural analysis confirmed that alkylation had indeed occurred at the carbonyl site (Fig. 1). The pyranoquinoline ring system in (2) was confirmed.

Hydrogen bonding occurs *via* C9—H9···O11^{i} [C9···O11^{i} = 3.367 (4) Å and C9—H9···O11^{i} = 148°; symmetry code: (i)

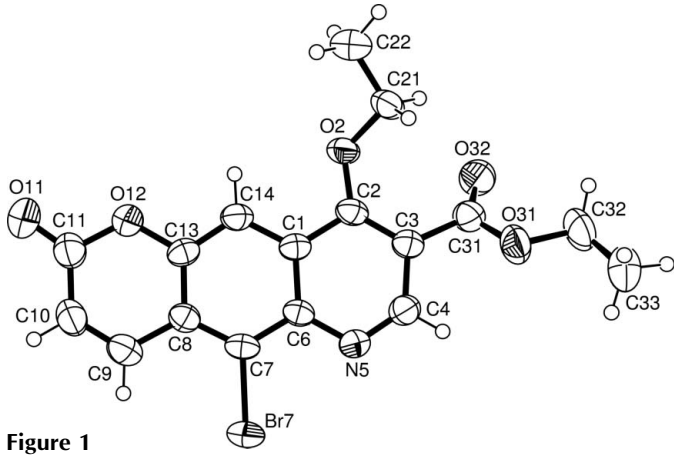


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

$1 - x, -y, z - \frac{1}{2}$], leading to $C(5)$ chains (Bernstein *et al.*, 1995) along [001] (Fig. 2).

The compound is isostructural with the chloro analogue (de Oliveira *et al.*, 2006).

Experimental

The title compound was obtained from the reaction between EtBr and (1) in DMF solution in the presence of K_2CO_3 (de Oliveira, 2003). Pure product (2) was obtained from the reaction mixture by column chromatography using hexane–ethyl acetate as the eluent (gradient 1:4 to 1:1). Crystals suitable for X-ray crystallography were grown from ethyl acetate (67% yield; m.p. 409–410 K).

Crystal data

$C_{17}H_{14}BrNO_5$
$M_r = 392.20$
Orthorhombic, $Pna2_1$
$a = 7.2542$ (8) Å
$b = 19.542$ (2) Å
$c = 11.2855$ (11) Å
$V = 1599.9$ (3) Å^3
$Z = 4$
$D_x = 1.628$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4263 reflections
$\theta = 3.5$–25.3°
$\mu = 2.60$ mm^{-1}
$T = 291$ (2) K
Prism, pale yellow
0.42 × 0.22 × 0.11 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.549$, $T_{max} = 0.752$
15681 measured reflections
5296 independent reflections
2737 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 32.5^\circ$
$h = -10 \rightarrow 10$
$k = -27 \rightarrow 29$
$l = -17 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.069$
$S = 0.80$
5296 reflections
219 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.034P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter: 0.013 (7)

All H atoms were located in difference maps and then treated as riding atoms, with C—H distances of 0.95 (aromatic) or 0.96 Å (methyl) and U_{iso}(H) values of $1.2U_{eq}$(C) (aromatic) or $1.5U_{eq}$(C) (methyl). *PLATON* (Spek, 2003) was used for the hydrogen-bonding analysis.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: CIFTAB (Sheldrick, 1997).

We thank the University of Aberdeen for funding of the X-ray diffractometer, and acknowledge the use of the EPSRC's Chemical Database Service at Daresbury Laboratory (Fletcher *et al.*, 1996).

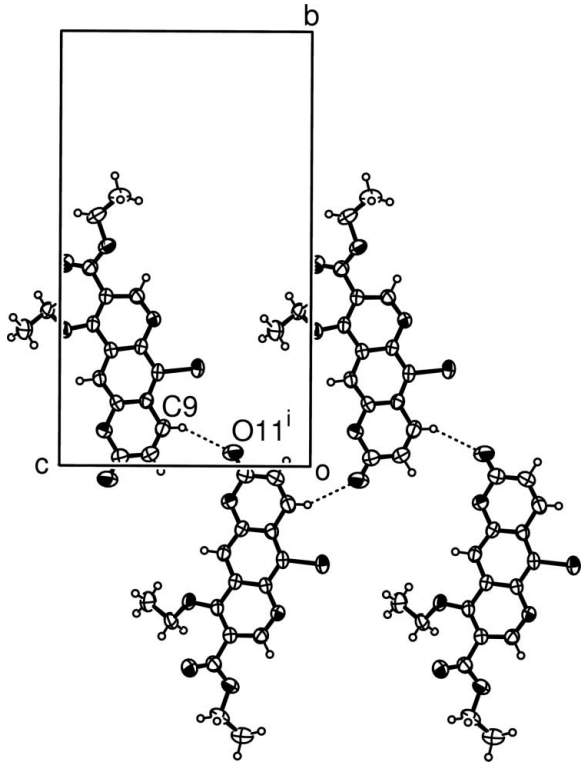


Figure 2
Part of the crystal structure of the title compound, showing the formation of a $C(5)$ chain (hydrogen bonds as dashed lines) along [001]. [Symmetry code: (i) $1 - x, -y, z - \frac{1}{2}$.]

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (1998). *SMART*. Version 5.054. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2000). *SADABS* (Version 2.03) and *SAINT* (Version 6.02a). Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
Matta, A. S. D. da, de Oliviera C. D. & Romeiro, G. A. (2000). *Heterocycl. Commun.* **6**, 511–514.
McArdle, P. (2003). *OSCAIL for Windows*. Version 10. Crystallography Centre, Chemistry Department, National University of Ireland, Galway, Ireland.
Oliveira, C. D. de (2003). PhD thesis, Universidade Federal Fluminense, Rio de Janeiro, Brazil.
Oliveira, C. D. de, Romeiro, G. A., Skakle, J. M. S., Wardell, J. L. & Wardell, S. M. S. V. (2006). *Acta Cryst.* E**62**, o1494–o1495.
Sheldrick, G. M. (1997). *SHELXS97*, *SHELXL97* and *CIFTAB*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **35**, 7–13.

supporting information

Acta Cryst. (2006). E**62**, o1492–o1493 [https://doi.org/10.1107/S1600536806007148]

Ethyl 5-bromo-9-ethoxy-2-oxo-2*H*-pyrano[2,3-*g*]quinoline-8-carboxylate

César D. de Oliveira, Gilberto A. Romeiro, Janet M. S. Skakle, James L. Wardell and Solange M. S. V. Wardell

Ethyl 5-bromo-9-ethoxy-2-oxo-2*H*-pyrano[2,3-*g*]quinoline-8-carboxylate

Crystal data

$C_{17}H_{14}BrNO_5$
$M_r = 392.20$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a = 7.2542$ (8) Å
$b = 19.542$ (2) Å
$c = 11.2855$ (11) Å
$V = 1599.9$ (3) Å^3
$Z = 4$
$F(000) = 792$
$D_x = 1.628$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4263 reflections
$\theta = 3.5$–$25.3°$
$\mu = 2.60$ mm^{-1}
$T = 291$ K
Prism, pale yellow
0.42 × 0.22 × 0.11 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2000)
$T_{min} = 0.549$, $T_{max} = 0.752$
15681 measured reflections
5296 independent reflections
2737 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 32.5°$, $\theta_{min} = 2.1°$
$h = -10 \rightarrow 10$
$k = -27 \rightarrow 29$
$l = -17 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.069$
$S = 0.80$
5296 reflections
219 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.034P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}
Absolute structure: Flack (1983)
Absolute structure parameter: 0.013 (7)

Special details

Experimental. IR (KBr, cm^{-1}): 2977, 1749, 1718, 1624, 1590, 1340, 1300. ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.51 (*t*, *J* = 7.2 Hz, 3H), 1.58 (*t*, *J* = 6.9 Hz, 3H), 4.43 (*q*, *J* = 6.9 Hz, 2H), 4.54 (*q*, *J* = 7.2 Hz, 2H), 6.89 (*d*, *J* = 9.9 Hz, 1H), 8.12 (*s*, 1H), 8.47 (*d*, *J* = 9.9 Hz, 1H), 9.19 (*s*, 1H). ^{13}C NMR (DMSO-d_6, 75 MHz): δ 14.1, 15.6, 61.9, 72.5, 108.7, 114.7, 120.7, 123.0, 15.1, 125.6, 142.1, 143.5, 150.7, 152.2, 158.7, 162.3, 164.4.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5465 (4)	0.26565 (12)	0.79440 (19)	0.0403 (5)
C2	0.5712 (4)	0.32561 (14)	0.8664 (2)	0.0431 (6)
O2	0.6179 (3)	0.31203 (10)	0.97852 (13)	0.0573 (5)
C21	0.7329 (4)	0.35662 (13)	1.0487 (2)	0.0532 (7)
H21A	0.6590	0.3919	1.0861	0.064*
H21B	0.8252	0.3784	0.9993	0.064*
C22	0.8232 (5)	0.31265 (16)	1.1406 (3)	0.0746 (8)
H22A	0.7308	0.2938	1.1920	0.112*
H22B	0.9076	0.3398	1.1862	0.112*
H22C	0.8890	0.2762	1.1024	0.112*
C3	0.5449 (4)	0.38935 (12)	0.8149 (2)	0.0444 (6)
C31	0.5426 (4)	0.45529 (14)	0.8793 (2)	0.0521 (7)
O31	0.6127 (3)	0.50489 (9)	0.81235 (17)	0.0669 (6)
O32	0.4762 (3)	0.46482 (10)	0.97594 (16)	0.0718 (6)
C32	0.5945 (5)	0.57525 (15)	0.8543 (3)	0.0793 (10)
H32A	0.6574	0.5808	0.9296	0.095*
H32B	0.4656	0.5869	0.8650	0.095*
C33	0.6788 (6)	0.61979 (16)	0.7632 (3)	0.0887 (11)
H33A	0.8093	0.6118	0.7606	0.133*
H33B	0.6559	0.6669	0.7826	0.133*
H33C	0.6260	0.6096	0.6872	0.133*
C4	0.4998 (4)	0.38994 (13)	0.6918 (2)	0.0476 (6)
H4	0.4916	0.4327	0.6559	0.057*
N5	0.4691 (3)	0.33752 (10)	0.62482 (17)	0.0463 (5)
C6	0.4915 (3)	0.27423 (11)	0.67461 (19)	0.0389 (5)
C7	0.4597 (3)	0.21492 (13)	0.6070 (2)	0.0432 (6)
Br7	0.38160 (3)	0.224825 (12)	0.44876 (3)	0.05827 (9)
C8	0.4855 (3)	0.14966 (13)	0.6517 (2)	0.0424 (5)
C9	0.4568 (4)	0.08662 (15)	0.5862 (2)	0.0555 (7)
H9	0.4138	0.0882	0.5086	0.067*
C10	0.4920 (4)	0.02688 (15)	0.6370 (3)	0.0621 (8)
H10	0.4719	−0.0128	0.5934	0.075*

C11	0.5597 (4)	0.02044 (16)	0.7560 (3)	0.0585 (7)
O11	0.6006 (4)	−0.03111 (11)	0.8062 (2)	0.0831 (7)
O12	0.5771 (3)	0.08092 (9)	0.81977 (15)	0.0539 (5)
C13	0.5460 (3)	0.14392 (12)	0.7696 (2)	0.0427 (6)
C14	0.5759 (3)	0.19997 (13)	0.8405 (2)	0.0447 (6)
H14	0.6153	0.1944	0.9183	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0370 (12)	0.0495 (15)	0.0342 (12)	−0.0018 (11)	−0.0001 (10)	0.0032 (10)
C2	0.0450 (16)	0.0529 (17)	0.0314 (13)	−0.0058 (12)	0.0022 (10)	−0.0016 (11)
O2	0.0836 (14)	0.0540 (10)	0.0344 (11)	−0.0195 (10)	−0.0123 (8)	0.0045 (7)
C21	0.067 (2)	0.0550 (16)	0.0381 (13)	−0.0098 (14)	−0.0046 (12)	−0.0074 (11)
C22	0.090 (2)	0.082 (2)	0.0516 (16)	−0.0017 (19)	−0.0200 (16)	0.0001 (15)
C3	0.0458 (14)	0.0496 (16)	0.0377 (13)	−0.0007 (12)	0.0012 (11)	−0.0010 (11)
C31	0.0584 (18)	0.0506 (18)	0.0473 (17)	0.0066 (13)	−0.0022 (13)	−0.0035 (13)
O31	0.0949 (16)	0.0458 (11)	0.0599 (12)	−0.0074 (11)	0.0144 (10)	−0.0113 (9)
O32	0.0991 (17)	0.0641 (12)	0.0522 (13)	0.0133 (12)	0.0118 (10)	−0.0052 (9)
C32	0.113 (3)	0.0495 (17)	0.075 (2)	−0.0023 (18)	0.007 (2)	−0.0234 (15)
C33	0.118 (3)	0.0493 (18)	0.099 (3)	−0.007 (2)	−0.004 (2)	−0.0037 (18)
C4	0.0507 (17)	0.0478 (15)	0.0444 (13)	0.0039 (12)	−0.0004 (11)	0.0029 (12)
N5	0.0532 (13)	0.0467 (13)	0.0390 (11)	0.0026 (10)	−0.0036 (10)	0.0027 (9)
C6	0.0360 (13)	0.0481 (14)	0.0326 (11)	−0.0006 (12)	0.0013 (9)	−0.0032 (11)
C7	0.0403 (13)	0.0601 (17)	0.0292 (11)	0.0006 (12)	0.0008 (9)	−0.0018 (10)
Br7	0.06948 (16)	0.06983 (14)	0.03550 (11)	−0.00117 (13)	−0.01183 (16)	−0.00210 (16)
C8	0.0418 (14)	0.0484 (15)	0.0370 (12)	−0.0008 (11)	0.0052 (10)	−0.0030 (10)
C9	0.0608 (17)	0.0620 (18)	0.0438 (15)	−0.0048 (15)	0.0020 (13)	−0.0078 (13)
C10	0.078 (2)	0.0468 (18)	0.0613 (18)	−0.0034 (16)	0.0012 (16)	−0.0107 (14)
C11	0.0665 (19)	0.0496 (17)	0.0595 (17)	−0.0031 (15)	0.0010 (15)	−0.0029 (14)
O11	0.115 (2)	0.0488 (13)	0.0849 (16)	0.0071 (13)	−0.0164 (13)	0.0045 (12)
O12	0.0656 (13)	0.0456 (10)	0.0505 (11)	−0.0022 (9)	−0.0056 (8)	0.0022 (8)
C13	0.0406 (13)	0.0495 (15)	0.0380 (13)	−0.0019 (11)	0.0012 (10)	0.0079 (11)
C14	0.0477 (16)	0.0516 (14)	0.0349 (12)	−0.0052 (12)	−0.0031 (10)	0.0038 (11)

Geometric parameters (Å, º)

C1—C14	1.401 (3)	C33—H33A	0.9600
C1—C6	1.419 (3)	C33—H33B	0.9600
C1—C2	1.437 (3)	C33—H33C	0.9600
C2—O2	1.336 (3)	C4—N5	1.292 (3)
C2—C3	1.388 (3)	C4—H4	0.9300
O2—C21	1.443 (3)	N5—C6	1.368 (3)
C21—C22	1.497 (4)	C6—C7	1.407 (3)
C21—H21A	0.9700	C7—C8	1.384 (3)
C21—H21B	0.9700	C7—Br7	1.883 (2)
C22—H22A	0.9600	C8—C13	1.406 (3)
C22—H22B	0.9600	C8—C9	1.452 (4)

C22—H22C	0.9600	C9—C10	1.326 (4)
C3—C4	1.427 (3)	C9—H9	0.9300
C3—C31	1.480 (3)	C10—C11	1.435 (4)
C31—O32	1.206 (3)	C10—H10	0.9300
C31—O31	1.330 (3)	C11—O11	1.193 (3)
O31—C32	1.460 (3)	C11—O12	1.390 (3)
C32—C33	1.480 (5)	O12—C13	1.374 (3)
C32—H32A	0.9700	C13—C14	1.373 (3)
C32—H32B	0.9700	C14—H14	0.9300

C14—C1—C6	120.3 (2)	H33A—C33—H33B	109.5
C14—C1—C2	121.2 (2)	C32—C33—H33C	109.5
C6—C1—C2	118.5 (2)	H33A—C33—H33C	109.5
O2—C2—C3	127.6 (2)	H33B—C33—H33C	109.5
O2—C2—C1	113.9 (2)	N5—C4—C3	127.0 (2)
C3—C2—C1	118.5 (2)	N5—C4—H4	116.5
C2—O2—C21	123.1 (2)	C3—C4—H4	116.5
O2—C21—C22	106.6 (2)	C4—N5—C6	117.1 (2)
O2—C21—H21A	110.4	N5—C6—C7	120.2 (2)
C22—C21—H21A	110.4	N5—C6—C1	122.1 (2)
O2—C21—H21B	110.4	C7—C6—C1	117.7 (2)
C22—C21—H21B	110.4	C8—C7—C6	122.6 (2)
H21A—C21—H21B	108.6	C8—C7—Br7	118.76 (18)
C21—C22—H22A	109.5	C6—C7—Br7	118.63 (18)
C21—C22—H22B	109.5	C7—C8—C13	117.4 (2)
H22A—C22—H22B	109.5	C7—C8—C9	125.2 (2)
C21—C22—H22C	109.5	C13—C8—C9	117.3 (2)
H22A—C22—H22C	109.5	C10—C9—C8	120.0 (3)
H22B—C22—H22C	109.5	C10—C9—H9	120.0
C2—C3—C4	116.5 (2)	C8—C9—H9	120.0
C2—C3—C31	125.2 (2)	C9—C10—C11	123.2 (3)
C4—C3—C31	118.0 (2)	C9—C10—H10	118.4
O32—C31—O31	123.6 (3)	C11—C10—H10	118.4
O32—C31—C3	125.7 (3)	O11—C11—O12	116.7 (3)
O31—C31—C3	110.5 (2)	O11—C11—C10	127.1 (3)
C31—O31—C32	117.9 (2)	O12—C11—C10	116.2 (3)
O31—C32—C33	106.9 (2)	C13—O12—C11	122.3 (2)
O31—C32—H32A	110.3	C14—C13—O12	116.7 (2)
C33—C32—H32A	110.3	C14—C13—C8	122.5 (2)
O31—C32—H32B	110.3	O12—C13—C8	120.9 (2)
C33—C32—H32B	110.3	C13—C14—C1	119.4 (2)
H32A—C32—H32B	108.6	C13—C14—H14	120.3
C32—C33—H33A	109.5	C1—C14—H14	120.3
C32—C33—H33B	109.5		

C14—C1—C2—O2	−1.9 (4)	C2—C1—C6—C7	−177.1 (2)
C6—C1—C2—O2	178.2 (2)	N5—C6—C7—C8	178.0 (2)
C14—C1—C2—C3	178.3 (2)	C1—C6—C7—C8	−2.1 (4)

C6—C1—C2—C3	−1.6 (4)	N5—C6—C7—Br7	−1.2 (3)
C3—C2—O2—C21	−31.6 (4)	C1—C6—C7—Br7	178.68 (18)
C1—C2—O2—C21	148.6 (2)	C6—C7—C8—C13	0.0 (4)
C2—O2—C21—C22	−154.4 (3)	Br7—C7—C8—C13	179.23 (18)
O2—C2—C3—C4	178.4 (3)	C6—C7—C8—C9	−179.4 (2)
C1—C2—C3—C4	−1.8 (4)	Br7—C7—C8—C9	−0.1 (4)
O2—C2—C3—C31	−7.6 (5)	C7—C8—C9—C10	177.7 (3)
C1—C2—C3—C31	172.2 (3)	C13—C8—C9—C10	−1.7 (4)
C2—C3—C31—O32	−38.1 (5)	C8—C9—C10—C11	−0.4 (5)
C4—C3—C31—O32	135.7 (3)	C9—C10—C11—O11	−177.1 (3)
C2—C3—C31—O31	146.4 (3)	C9—C10—C11—O12	3.7 (4)
C4—C3—C31—O31	−39.7 (4)	O11—C11—O12—C13	175.6 (3)
O32—C31—O31—C32	−5.1 (4)	C10—C11—O12—C13	−5.1 (4)
C3—C31—O31—C32	170.5 (3)	C11—O12—C13—C14	−177.1 (2)
C31—O31—C32—C33	−178.7 (3)	C11—O12—C13—C8	3.3 (4)
C2—C3—C4—N5	4.9 (4)	C7—C8—C13—C14	1.3 (4)
C31—C3—C4—N5	−169.5 (3)	C9—C8—C13—C14	−179.3 (2)
C3—C4—N5—C6	−3.8 (4)	C7—C8—C13—O12	−179.1 (2)
C4—N5—C6—C7	179.7 (2)	C9—C8—C13—O12	0.3 (4)
C4—N5—C6—C1	−0.2 (4)	O12—C13—C14—C1	−179.9 (2)
C14—C1—C6—N5	−177.1 (2)	C8—C13—C14—C1	−0.4 (4)
C2—C1—C6—N5	2.8 (4)	C6—C1—C14—C13	−1.8 (4)
C14—C1—C6—C7	3.0 (4)	C2—C1—C14—C13	178.3 (2)