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(4*R*)-4-Benzyl-3-[(4*S*)-4-chloro-4-[(*S*)-2,2-dimethyl-1,3-dioxolan-4-yl]-butanoyl]-1,3-oxazolidin-2-one**Sandra Börding, Carsten Strohmann, Hans Preut* and Martin Hiersemann**

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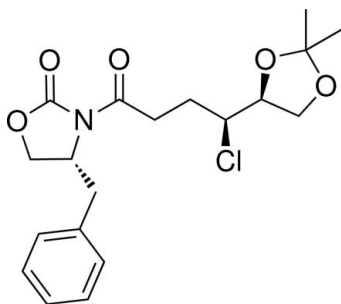
Received 28 November 2011; accepted 14 December 2011

Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.039; wR factor = 0.047; data-to-parameter ratio = 15.2.

The title compound, $\text{C}_{19}\text{H}_{24}\text{ClNO}_5$, was synthesized and subsequently employed in an Evans alkylation. The purpose was to prove the absolute configuration in the projected synthesis of the side chain of (–)-Lytophilippine A. The oxazolidinone and the isopropylidene acetal rings have twisted conformations. The oxazolidinone and side-chain carbonyl groups are orientated in an antiperiplanar arrangement to minimize van der Waals repulsions. Furthermore, the Cl atom and the acetone-protected secondary alcohol are also in an antiperiplanar arrangement with a torsion angle of $173.64(14)^\circ$. The absolute configuration was determined and agrees with the configuration of the used chiral auxiliary.

Related literature

For background to the synthesis, see: Gille & Hiersemann (2010); Jang *et al.* (2011); Řezanka *et al.* (2004). For Evans alkylation, see: Evans *et al.* (1981, 1982).

**Experimental***Crystal data*

$\text{C}_{19}\text{H}_{24}\text{ClNO}_5$
 $M_r = 381.84$
 Monoclinic, $P2_1$
 $a = 11.7552(9)$ Å
 $b = 5.9139(4)$ Å
 $c = 13.8789(11)$ Å
 $\beta = 109.023(9)^\circ$

$V = 912.16(12)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.24$ mm⁻¹
 $T = 173$ K
 $0.40 \times 0.20 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur
 Sapphire3 diffractometer
 Absorption correction: multi-scan
 (*CrysAlis RED*; Oxford
 Diffraction, 2008)
 $T_{\min} = 0.910$, $T_{\max} = 0.954$

6562 measured reflections
 3594 independent reflections
 2515 reflections with $I > 2s(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.047$
 $S = 0.98$
 3594 reflections
 237 parameters
 1 restraint

H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³
 Absolute structure: Flack (1983),
 1198 Friedel pairs
 Flack parameter: 0.11 (5)

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL-Plus* (Sheldrick, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL-Plus*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FF2045).

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supplementary materials

Acta Cryst. (2012). E68, o169 [doi:10.1107/S1600536811053840]

(4*R*)-4-Benzyl-3-{(4*S*)-4-chloro-4-[(*S*)-2,2-dimethyl-1,3-dioxolan-4-yl]butanoyl}-1,3-oxazolidin-2-one

S. Börding, C. Strohmann, H. Preut and M. Hiersemann

Comment

The title compound I was obtained during the synthesis of the side chain of (–)-Lytophilippine A (Řezanka *et al.*, 2004). Recently our research group published the synthesis of the core fragment of (–)-Lytophilippine A (Gille & Hiersemann, 2010). Shortly after, the first total synthesis of the postulated structure was published (Jang *et al.*, 2011). Compound I was synthesized and subsequently applied in an Evans alkylation (Evans *et al.*, 1981; Evans *et al.*, 1982) to install the stereogenic center at C23. The synthesis of I was carried out from carboxylic acid and (*R*)-4-benzyloxazolidin-2-one.

The oxazolidinone ring adopts a nearly coplanar conformation and the isopropylidene acetal is an open envelope-like structure. The oxazolidinone- and side-chain carbonyl groups are orientated in an antiperiplanar arrangement to minimize van der Waals repulsions. The dihedral angle between the plane through N, C10, O2 and the plane through N, C11, O3 is 9.4 (4)°. Furthermore, the chlorine atom and the acetonide protected secondary alcohol are also in an antiperiplanar arrangement with a torsion angle of 173.64 (14)°. The absolute configuration was determined and agrees with the configuration of the used chiral auxiliary.

Experimental

To a solution of carboxylic acid (840 mg, 3.77 mmol, 1.0 eq) in THF (20 ml, 5 ml/mmol) was added Et₃N (1.14 ml, 8.15 mmol, 2.0 eq) and pivaloylchloride (0.6 ml, 4.89 mmol, 1.2 eq) at 253 K. After stirring at this temperature for 2 h the mixture was warmed to 273 K. Solid LiCl (240 mg, 5.67 mmol, 1.5 eq) and (*R*)-4-benzyloxazolidin-2-one (669 mg, 3.77 mmol, 1.0 eq) were added. The reaction was quenched with H₂O after stirring for 2 h at room temperature. The layers were separated and the aqueous phase extracted with Et₂O. The combined organic phases were dried over anhydrous MgSO₄, filtered and concentrated under reduced pressure. Flash column chromatography (cyclohexane/ethyl acetate 5/1) afforded the title compound (1.12 g, 2.93 mmol, 84%) as a thick colourless oil. Single crystals of (I) were obtained by crystallization from isohexane to provide white needles. *R*_f 0.43 (cyclohexane/ethyl acetate 2/1); Anal. Calcd. for C₁₉H₂₄ClNO₅: C, 59.8; H, 6.3; N, 3.7; Found: C, 59.8; H, 6.5; N, 3.6; [*α*]_D²⁰ -51.4 (c 1.02, CH₃Cl); *M* = 381.85 g/mol.

Refinement

The hydrogen atoms were placed in calculated positions with C–H bond distances in the range from 0.95 to 1.00 Å and refined as riding on their parent atoms with *U*_{iso} = 1.2 or 1.5 × *U*_{eq}(C).

Figures

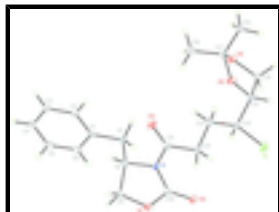


Fig. 1. The molecular structure of the title compound, showing the labelling of all non-H atoms. Displacement ellipsoids are shown at the 30% probability level.

(4R)-4-Benzyl-3-((4S)-4-chloro-4-[(S)-2,2-dimethyl-1,3-dioxolan-4-yl]butanoyl)-1,3-oxazolidin-2-one

Crystal data

$C_{19}H_{24}ClNO_5$

$M_r = 381.84$

Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 11.7552$ (9) Å

$b = 5.9139$ (4) Å

$c = 13.8789$ (11) Å

$\beta = 109.023$ (9)°

$V = 912.16$ (12) Å³

$Z = 2$

$F(000) = 404$

$D_x = 1.390$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2793 reflections

$\theta = 2.8$ – 29.0 °

$\mu = 0.24$ mm⁻¹

$T = 173$ K

Block, white

$0.40 \times 0.20 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer

Radiation source: Enhance (Mo) X-ray Source graphite

Detector resolution: 16.0560 pixels mm⁻¹

ω scans

Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)

$T_{\min} = 0.910$, $T_{\max} = 0.954$

6562 measured reflections

3594 independent reflections

2515 reflections with $I > 2s(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 26.0$ °, $\theta_{\min} = 2.8$ °

$h = -14 \rightarrow 14$

$k = -7 \rightarrow 7$

$l = -17 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.047$

$S = 0.98$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.007P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

3594 reflections	$\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$
237 parameters	$\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$
1 restraint	Absolute structure: Flack (1983), 1198 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.11 (5)

Special details

Experimental. CrysAlis RED (Oxford Diffraction, 2008), Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1657 (2)	-0.3801 (4)	0.17220 (18)	0.0189 (6)
C2	0.0920 (2)	-0.2107 (4)	0.18929 (18)	0.0224 (7)
H2	0.1267	-0.0727	0.2202	0.027*
C3	-0.0317 (2)	-0.2418 (4)	0.1616 (2)	0.0290 (7)
H3	-0.0810	-0.1269	0.1750	0.035*
C4	-0.0827 (2)	-0.4398 (4)	0.11451 (19)	0.0270 (7)
H4	-0.1675	-0.4598	0.0940	0.032*
C5	-0.0110 (2)	-0.6085 (4)	0.0972 (2)	0.0285 (7)
H5	-0.0459	-0.7454	0.0653	0.034*
C6	0.1125 (2)	-0.5773 (4)	0.12673 (19)	0.0245 (7)
H6	0.1616	-0.6951	0.1153	0.029*
C7	0.30014 (18)	-0.3420 (4)	0.20129 (17)	0.0201 (6)
H7A	0.3425	-0.4887	0.2185	0.024*
H7B	0.3273	-0.2439	0.2623	0.024*
C8	0.3321 (2)	-0.2315 (4)	0.11426 (18)	0.0191 (6)
H8	0.2796	-0.0967	0.0885	0.023*
C9	0.3254 (2)	-0.3926 (4)	0.02582 (19)	0.0312 (7)
H9A	0.2762	-0.5272	0.0279	0.037*
H9B	0.2896	-0.3155	-0.0405	0.037*
C10	0.5254 (2)	-0.3120 (5)	0.10476 (17)	0.0206 (6)
C11	0.4985 (2)	0.0280 (4)	0.20304 (19)	0.0216 (6)
C12	0.6276 (2)	0.0996 (4)	0.2280 (2)	0.0244 (7)
H12A	0.6385	0.1773	0.1684	0.029*
H12B	0.6800	-0.0358	0.2430	0.029*
C13	0.6643 (2)	0.2563 (4)	0.31873 (19)	0.0220 (7)

supplementary materials

H13A	0.6713	0.1670	0.3808	0.026*
H13B	0.5995	0.3689	0.3106	0.026*
C14	0.7817 (2)	0.3812 (4)	0.33466 (19)	0.0209 (6)
H14	0.7708	0.4861	0.2758	0.025*
C15	0.8252 (2)	0.5187 (4)	0.43295 (18)	0.0209 (6)
H15	0.9058	0.5866	0.4413	0.025*
C16	0.8292 (2)	0.3846 (4)	0.52813 (18)	0.0244 (6)
H16A	0.8405	0.2212	0.5187	0.029*
H16B	0.8949	0.4389	0.5886	0.029*
C17	0.6855 (2)	0.6563 (4)	0.50682 (18)	0.0229 (6)
C18	0.55163 (18)	0.6806 (5)	0.46026 (17)	0.0278 (6)
H18A	0.5204	0.5595	0.4104	0.042*
H18B	0.5328	0.8275	0.4261	0.042*
H18C	0.5143	0.6706	0.5138	0.042*
C19	0.7398 (2)	0.8166 (4)	0.59485 (18)	0.0272 (7)
H19A	0.7155	0.7708	0.6531	0.041*
H19B	0.7114	0.9706	0.5743	0.041*
H19C	0.8277	0.8121	0.6141	0.041*
Cl	0.90160 (5)	0.18577 (11)	0.33911 (5)	0.03519 (19)
N	0.46002 (17)	-0.1656 (3)	0.14456 (15)	0.0161 (5)
O1	0.44892 (15)	-0.4571 (3)	0.04026 (13)	0.0265 (4)
O2	0.63229 (13)	-0.3207 (3)	0.12130 (12)	0.0242 (4)
O3	0.42567 (14)	0.1337 (3)	0.22968 (13)	0.0314 (5)
O4	0.71441 (14)	0.4278 (2)	0.53829 (13)	0.0204 (4)
O5	0.73891 (12)	0.6923 (3)	0.42756 (11)	0.0231 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0209 (14)	0.0224 (16)	0.0130 (15)	-0.0011 (12)	0.0049 (13)	0.0024 (11)
C2	0.0283 (16)	0.0211 (14)	0.0183 (17)	0.0020 (13)	0.0085 (15)	-0.0011 (12)
C3	0.0288 (17)	0.0318 (17)	0.0305 (19)	0.0072 (14)	0.0151 (16)	-0.0004 (14)
C4	0.0206 (16)	0.0364 (16)	0.0244 (18)	-0.0029 (14)	0.0079 (15)	0.0005 (14)
C5	0.0306 (17)	0.0216 (16)	0.0353 (19)	-0.0098 (14)	0.0132 (16)	-0.0060 (14)
C6	0.0290 (17)	0.0195 (14)	0.0296 (18)	0.0021 (13)	0.0158 (15)	0.0014 (13)
C7	0.0225 (13)	0.0186 (14)	0.0183 (15)	-0.0005 (14)	0.0054 (12)	-0.0032 (12)
C8	0.0156 (14)	0.0228 (14)	0.0176 (16)	0.0021 (11)	0.0038 (13)	-0.0029 (12)
C9	0.0176 (14)	0.0477 (19)	0.0294 (18)	-0.0047 (14)	0.0094 (15)	-0.0112 (14)
C10	0.0278 (14)	0.0202 (13)	0.0131 (14)	-0.0007 (16)	0.0058 (13)	0.0073 (14)
C11	0.0259 (17)	0.0204 (14)	0.0168 (16)	-0.0011 (13)	0.0046 (14)	0.0036 (13)
C12	0.0197 (14)	0.0233 (15)	0.0334 (18)	-0.0028 (12)	0.0132 (14)	-0.0045 (12)
C13	0.0198 (14)	0.0239 (16)	0.0230 (17)	0.0007 (12)	0.0078 (14)	-0.0014 (12)
C14	0.0198 (15)	0.0208 (14)	0.0239 (17)	0.0079 (13)	0.0097 (14)	0.0060 (13)
C15	0.0176 (15)	0.0202 (14)	0.0242 (17)	-0.0038 (12)	0.0058 (14)	-0.0033 (13)
C16	0.0222 (16)	0.0280 (15)	0.0205 (17)	-0.0031 (13)	0.0033 (14)	-0.0005 (13)
C17	0.0277 (14)	0.0224 (15)	0.0215 (16)	-0.0027 (15)	0.0122 (14)	-0.0001 (14)
C18	0.0277 (14)	0.0233 (13)	0.0337 (17)	-0.0015 (16)	0.0116 (14)	0.0011 (15)
C19	0.0309 (17)	0.0260 (15)	0.0279 (18)	-0.0057 (12)	0.0140 (16)	-0.0055 (13)

Cl	0.0234 (4)	0.0399 (4)	0.0430 (5)	0.0074 (4)	0.0119 (4)	-0.0058 (4)
N	0.0116 (12)	0.0175 (12)	0.0205 (14)	0.0006 (9)	0.0070 (11)	-0.0043 (9)
O1	0.0241 (11)	0.0301 (10)	0.0254 (12)	-0.0011 (8)	0.0083 (10)	-0.0127 (9)
O2	0.0193 (8)	0.0274 (9)	0.0273 (11)	0.0067 (10)	0.0096 (8)	-0.0029 (10)
O3	0.0252 (9)	0.0262 (11)	0.0473 (14)	-0.0023 (9)	0.0182 (10)	-0.0133 (9)
O4	0.0215 (11)	0.0197 (9)	0.0234 (11)	0.0000 (9)	0.0120 (9)	0.0040 (8)
O5	0.0296 (10)	0.0209 (9)	0.0241 (10)	0.0017 (10)	0.0158 (9)	0.0022 (10)

Geometric parameters (Å, °)

C1—C6	1.376 (3)	C11—C12	1.503 (3)
C1—C2	1.394 (3)	C12—C13	1.508 (3)
C1—C7	1.515 (3)	C12—H12A	0.9900
C2—C3	1.390 (3)	C12—H12B	0.9900
C2—H2	0.9500	C13—C14	1.517 (3)
C3—C4	1.379 (3)	C13—H13A	0.9900
C3—H3	0.9500	C13—H13B	0.9900
C4—C5	1.377 (3)	C14—C15	1.526 (3)
C4—H4	0.9500	C14—Cl	1.808 (2)
C5—C6	1.386 (3)	C14—H14	1.0000
C5—H5	0.9500	C15—O5	1.428 (2)
C6—H6	0.9500	C15—C16	1.528 (3)
C7—C8	1.524 (3)	C15—H15	1.0000
C7—H7A	0.9900	C16—O4	1.425 (3)
C7—H7B	0.9900	C16—H16A	0.9900
C8—N	1.476 (3)	C16—H16B	0.9900
C8—C9	1.536 (3)	C17—O4	1.426 (3)
C8—H8	1.0000	C17—O5	1.449 (2)
C9—O1	1.450 (2)	C17—C18	1.500 (3)
C9—H9A	0.9900	C17—C19	1.514 (3)
C9—H9B	0.9900	C18—H18A	0.9800
C10—O2	1.202 (2)	C18—H18B	0.9800
C10—O1	1.350 (3)	C18—H18C	0.9800
C10—N	1.386 (3)	C19—H19A	0.9800
C11—O3	1.211 (2)	C19—H19B	0.9800
C11—N	1.391 (3)	C19—H19C	0.9800
C6—C1—C2	118.2 (2)	C12—C13—C14	114.81 (19)
C6—C1—C7	121.8 (2)	C12—C13—H13A	108.6
C2—C1—C7	120.0 (2)	C14—C13—H13A	108.6
C3—C2—C1	120.7 (2)	C12—C13—H13B	108.6
C3—C2—H2	119.7	C14—C13—H13B	108.6
C1—C2—H2	119.7	H13A—C13—H13B	107.5
C4—C3—C2	119.8 (2)	C13—C14—C15	114.5 (2)
C4—C3—H3	120.1	C13—C14—Cl	110.85 (15)
C2—C3—H3	120.1	C15—C14—Cl	106.22 (17)
C5—C4—C3	120.1 (3)	C13—C14—H14	108.4
C5—C4—H4	120.0	C15—C14—H14	108.4
C3—C4—H4	120.0	Cl—C14—H14	108.4
C4—C5—C6	119.6 (2)	O5—C15—C14	108.09 (19)

supplementary materials

C4—C5—H5	120.2	O5—C15—C16	103.84 (17)
C6—C5—H5	120.2	C14—C15—C16	113.78 (18)
C1—C6—C5	121.6 (2)	O5—C15—H15	110.3
C1—C6—H6	119.2	C14—C15—H15	110.3
C5—C6—H6	119.2	C16—C15—H15	110.3
C1—C7—C8	110.94 (19)	O4—C16—C15	103.26 (18)
C1—C7—H7A	109.5	O4—C16—H16A	111.1
C8—C7—H7A	109.5	C15—C16—H16A	111.1
C1—C7—H7B	109.5	O4—C16—H16B	111.1
C8—C7—H7B	109.5	C15—C16—H16B	111.1
H7A—C7—H7B	108.0	H16A—C16—H16B	109.1
N—C8—C7	112.25 (19)	O4—C17—O5	104.61 (18)
N—C8—C9	99.97 (18)	O4—C17—C18	109.5 (2)
C7—C8—C9	113.96 (18)	O5—C17—C18	108.19 (19)
N—C8—H8	110.1	O4—C17—C19	110.5 (2)
C7—C8—H8	110.1	O5—C17—C19	110.32 (19)
C9—C8—H8	110.1	C18—C17—C19	113.3 (2)
O1—C9—C8	105.4 (2)	C17—C18—H18A	109.5
O1—C9—H9A	110.7	C17—C18—H18B	109.5
C8—C9—H9A	110.7	H18A—C18—H18B	109.5
O1—C9—H9B	110.7	C17—C18—H18C	109.5
C8—C9—H9B	110.7	H18A—C18—H18C	109.5
H9A—C9—H9B	108.8	H18B—C18—H18C	109.5
O2—C10—O1	121.9 (3)	C17—C19—H19A	109.5
O2—C10—N	129.2 (3)	C17—C19—H19B	109.5
O1—C10—N	108.97 (19)	H19A—C19—H19B	109.5
O3—C11—N	118.3 (2)	C17—C19—H19C	109.5
O3—C11—C12	123.1 (2)	H19A—C19—H19C	109.5
N—C11—C12	118.6 (2)	H19B—C19—H19C	109.5
C11—C12—C13	110.96 (19)	C10—N—C11	129.0 (2)
C11—C12—H12A	109.4	C10—N—C8	111.57 (19)
C13—C12—H12A	109.4	C11—N—C8	119.42 (19)
C11—C12—H12B	109.4	C10—O1—C9	110.18 (18)
C13—C12—H12B	109.4	C16—O4—C17	106.18 (16)
H12A—C12—H12B	108.0	C15—O5—C17	109.33 (17)
C6—C1—C2—C3	0.4 (4)	O2—C10—N—C11	9.7 (4)
C7—C1—C2—C3	179.0 (2)	O1—C10—N—C11	-170.7 (2)
C1—C2—C3—C4	-1.5 (4)	O2—C10—N—C8	-172.6 (3)
C2—C3—C4—C5	1.5 (4)	O1—C10—N—C8	6.9 (3)
C3—C4—C5—C6	-0.4 (4)	O3—C11—N—C10	179.5 (2)
C2—C1—C6—C5	0.7 (4)	C12—C11—N—C10	0.9 (3)
C7—C1—C6—C5	-177.9 (2)	O3—C11—N—C8	2.0 (3)
C4—C5—C6—C1	-0.7 (4)	C12—C11—N—C8	-176.5 (2)
C6—C1—C7—C8	90.3 (3)	C7—C8—N—C10	104.9 (2)
C2—C1—C7—C8	-88.2 (3)	C9—C8—N—C10	-16.3 (2)
C1—C7—C8—N	172.7 (2)	C7—C8—N—C11	-77.2 (3)
C1—C7—C8—C9	-74.6 (3)	C9—C8—N—C11	161.60 (19)
N—C8—C9—O1	19.2 (2)	O2—C10—O1—C9	-173.6 (2)
C7—C8—C9—O1	-100.7 (2)	N—C10—O1—C9	6.8 (3)

supplementary materials

O3—C11—C12—C13	21.4 (3)	C8—C9—O1—C10	-17.1 (2)
N—C11—C12—C13	-160.2 (2)	C15—C16—O4—C17	-35.9 (2)
C11—C12—C13—C14	-166.14 (19)	O5—C17—O4—C16	33.2 (2)
C12—C13—C14—C15	-173.5 (2)	C18—C17—O4—C16	148.95 (19)
C12—C13—C14—Cl	-53.4 (2)	C19—C17—O4—C16	-85.6 (2)
C13—C14—C15—O5	-63.7 (2)	C14—C15—O5—C17	116.1 (2)
Cl—C14—C15—O5	173.64 (14)	C16—C15—O5—C17	-5.0 (2)
C13—C14—C15—C16	51.1 (3)	O4—C17—O5—C15	-16.6 (2)
Cl—C14—C15—C16	-71.6 (2)	C18—C17—O5—C15	-133.4 (2)
O5—C15—C16—O4	24.8 (2)	C19—C17—O5—C15	102.2 (2)
C14—C15—C16—O4	-92.5 (2)		

Fig. 1

