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[2-(4-Chlorophenyl)-5-phenyloxolan-3-yl](cyclopentenyl)methanone

Jae Kyun Lee,[a] Satish N. Chavre,[a] Yong Seo Cho,[a] Yeonhee Lee[b] and Joo Hwan Cha[b]*

[a]Center for Neuro-Medicine, Korea Institute of Science & Technology, Hwarangro 14-gil, Seongbuk-gu, Seoul 136-791, Republic of Korea, and [b]Advanced Analysis Center, Korea Institute of Science & Technology, Hwarangro 14-gil, Seongbuk-gu, Seoul 136-791, Republic of Korea
Correspondence e-mail: jhcha@kist.re.kr

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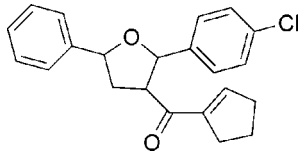
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.051; wR factor = 0.139; data-to-parameter ratio = 14.6.

In the title compound, $C_{22}H_{21}ClO_2$, the oxolane ring adopts a twisted conformation. The dihedral angles between the mean plane of the oxolane ring and the mean planes of the 4-chlorophenyl, phenyl and cyclopentenyl rings are 71.81 (18), 76.9 (18) and 82.08 (18)°, respectively.

Related literature

For general background to the Prins-type cyclization for the synthesis of oxolanes, see: Chavre *et al.* (2006, 2008); Cohen *et al.* (2001); Shin *et al.* (2005).



Experimental

Crystal data

$C_{22}H_{21}ClO_2$
M_r = 352.86
Monoclinic, $P2_1/n$
a = 5.7575 (6) Å
b = 11.3547 (12) Å
c = 28.554 (3) Å
β = 94.442 (8)°
V = 1861.1 (4) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 1.90 mm^{-1}
T = 296 K
0.50 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Rigaku, 1995)
T_{min} = 0.301, T_{max} = 0.684
18544 measured reflections
3363 independent reflections
2012 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.073

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.139
S = 1.05
3363 reflections
230 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Data collection: *RAPID-AUTO* (Rigaku, 2006); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SIR2008* in *Il Milione* (Burla *et al.*, 2007); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2010); software used to prepare material for publication: *CrystalStructure*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GO2037).

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[2-(4-Chlorophenyl)-5-phenyloxolan-3-yl](cyclopentenyl)methanone

Jae Kyun Lee, Satish N. Chavre, Yong Seo Cho, Yeonhee Lee and Joo Hwan Cha

S1. Comment

The oxolane moiety is an important heterocycle constituent in many bioactive natural products. New synthetic methodologies for 2,5-disubstituted oxolanes having an allenyl group, *via* Prins-type cyclization, has been subject of development since last decade (Shin *et al.*, 2005). Cohen and colleagues synthesized the polysubstituted oxolane by Prins-Pinacol reaction (Cohen *et al.*, 2001). 5-Exocyclic products, 2,3,5-trisustituted oxolanes, were synthesized from homopropargylic alcohols with alkynes and aldehydes (Chavre *et al.*, 2006, 2008).

In the molecule of Fig. 1, the oxolane ring adopts a twisted conformation on C5–O1. The dihedral angle between the chlorophenyl and cyclopentenyl rings is 31.03 (17)°.

S2. Experimental

To a stirred solution of 1-(4-hydroxy-4-phenylbut-1-ynyl)cyclopentanol (0.31 mmol) and 4-Chlorobenzaldehyde (0.37 mmol) in dry diethyl ether (3.0 ml) was added slowly TMSOTf (0.93 mmol) for 10 min and stirred for 1 h at -78°C. The mixture was allowed to warm to room temperature slowly for 3 h. The mixture was stirred at room temperature for additional 1–2 h until the completion of reaction. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ and diluted with 10 ml of diethyl ether. The organic solution was washed with water and brine, and the organic layer was dried over $MgSO_4$, filtered, concentrated and purification by silica gel column chromatography to afford the title compound (54%) as a yellow crystal.

S3. Refinement

All hydrogen atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.98 Å and *U*iso(H) = 1.2 or 1.5 *U*eq(C). Rotating group model was applied for the methyl groups.

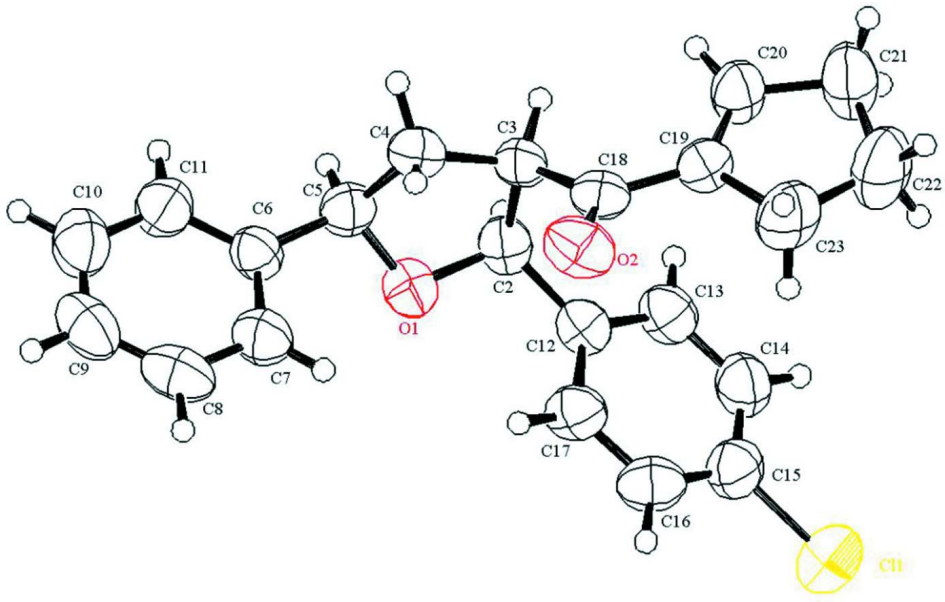

Figure 1

The structure of the title compound, with displacement ellipsoids drawn at the 50% probability level and H atoms with arbitrary radius.

[2-(4-Chlorophenyl)-5-phenyloxolan-3-yl](cyclopentenyl)methanone

Crystal data

$C_{22}H_{21}ClO_2$
$M_r = 352.86$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 5.7575\ (6)$ Å
$b = 11.3547\ (12)$ Å
$c = 28.554\ (3)$ Å
$\beta = 94.442\ (8)°$
$V = 1861.1\ (4)$ Å^3
$Z = 4$
$F(000) = 744.00$
$D_x = 1.259$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54187$ Å
Cell parameters from 12997 reflections
$\theta = 3.1–68.3°$
$\mu = 1.90$ mm^{-1}
$T = 296$ K
Needle, yellow
0.50 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*ABSCOR*; Rigaku, 1995)
$T_{min} = 0.301$, $T_{max} = 0.684$
18544 measured reflections
3363 independent reflections
2012 reflections with $F^2 > 2\sigma(F^2)$
$R_{int} = 0.073$
$\theta_{max} = 68.2°$
$h = -6\rightarrow6$
$k = -13\rightarrow13$
$l = -34\rightarrow34$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.139$
$S = 1.05$
3363 reflections
230 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.1808P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

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 was performed using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F^2. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\ \sigma(F^2)$ is used only for calculating *R*-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl(1)	0.34139 (17)	0.11812 (7)	0.53247 (3)	0.1065 (4)
O(1)	0.2994 (3)	0.37307 (13)	0.74183 (5)	0.0597 (5)
O(2)	0.6450 (3)	0.53421 (16)	0.66245 (6)	0.0763 (6)
C(2)	0.1844 (5)	0.4198 (2)	0.70022 (8)	0.0574 (7)
C(3)	0.2806 (5)	0.5488 (2)	0.69627 (8)	0.0561 (7)
C(4)	0.4071 (5)	0.5703 (3)	0.74448 (8)	0.0700 (8)
C(5)	0.3328 (5)	0.4685 (3)	0.77457 (8)	0.0602 (7)
C(6)	0.5050 (5)	0.4344 (2)	0.81393 (8)	0.0541 (6)
C(7)	0.7050 (5)	0.3738 (3)	0.80578 (10)	0.0682 (8)
C(8)	0.8695 (5)	0.3488 (3)	0.84223 (12)	0.0802 (9)
C(9)	0.8361 (6)	0.3839 (3)	0.88696 (11)	0.0805 (9)
C(10)	0.6385 (6)	0.4435 (3)	0.89575 (9)	0.0795 (9)
C(11)	0.4730 (5)	0.4682 (3)	0.85944 (9)	0.0668 (7)
C(12)	0.2209 (5)	0.3392 (2)	0.65941 (8)	0.0526 (6)
C(13)	0.0550 (5)	0.3341 (3)	0.62177 (9)	0.0628 (7)
C(14)	0.0916 (5)	0.2667 (3)	0.58285 (9)	0.0713 (8)
C(15)	0.2948 (6)	0.2045 (3)	0.58140 (9)	0.0680 (8)
C(16)	0.4618 (5)	0.2066 (3)	0.61850 (10)	0.0697 (8)
C(17)	0.4230 (5)	0.2737 (2)	0.65745 (9)	0.0632 (7)
C(18)	0.4382 (5)	0.5586 (2)	0.65654 (8)	0.0549 (6)
C(19)	0.3382 (5)	0.5935 (2)	0.60977 (8)	0.0523 (6)
C(20)	0.1393 (5)	0.6481 (3)	0.59902 (9)	0.0667 (8)
C(21)	0.1054 (6)	0.6755 (3)	0.54765 (9)	0.0881 (10)
C(22)	0.3045 (6)	0.6163 (4)	0.52732 (10)	0.0999 (11)
C(23)	0.4688 (5)	0.5744 (3)	0.56742 (9)	0.0829 (9)
H(2)	0.0171	0.4240	0.7043	0.0689*
H(3)	0.1505	0.6041	0.6912	0.0673*
H(4A)	0.5746	0.5702	0.7426	0.0840*
H(4B)	0.3612	0.6451	0.7573	0.0840*
H(5)	0.1839	0.4877	0.7872	0.0723*
H(7)	0.7295	0.3495	0.7755	0.0818*

H(8)	1.0039	0.3077	0.8363	0.0963*
H(9)	0.9478	0.3673	0.9113	0.0966*
H(10)	0.6151	0.4674	0.9262	0.0954*
H(11)	0.3380	0.5083	0.8657	0.0802*
H(13)	−0.0827	0.3765	0.6227	0.0753*
H(14)	−0.0211	0.2635	0.5577	0.0855*
H(16)	0.5987	0.1636	0.6174	0.0836*
H(17)	0.5345	0.2749	0.6828	0.0758*
H(21A)	−0.0419	0.6444	0.5342	0.1057*
H(21B)	0.1090	0.7598	0.5423	0.1057*
H(22A)	0.3833	0.6710	0.5078	0.1199*
H(22B)	0.2495	0.5501	0.5081	0.1199*
H(23A)	0.5063	0.4918	0.5638	0.0994*
H(23B)	0.6120	0.6198	0.5694	0.0994*
H(20)	0.033 (5)	0.672 (3)	0.6215 (9)	0.086 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl(1)	0.1507 (9)	0.0956 (7)	0.0744 (5)	0.0158 (6)	0.0156 (5)	−0.0156 (4)
O(1)	0.0594 (12)	0.0608 (11)	0.0580 (10)	−0.0031 (9)	−0.0002 (8)	0.0051 (8)
O(2)	0.0438 (12)	0.0850 (14)	0.1001 (14)	0.0143 (10)	0.0048 (10)	0.0100 (11)
C(2)	0.0435 (15)	0.0749 (17)	0.0541 (14)	0.0051 (13)	0.0051 (12)	0.0053 (12)
C(3)	0.0554 (16)	0.0592 (15)	0.0541 (14)	0.0195 (13)	0.0067 (12)	0.0033 (11)
C(4)	0.091 (2)	0.0526 (15)	0.0655 (16)	0.0113 (15)	−0.0014 (15)	−0.0010 (12)
C(5)	0.0540 (17)	0.0693 (17)	0.0580 (15)	0.0132 (13)	0.0085 (13)	−0.0031 (13)
C(6)	0.0498 (16)	0.0577 (14)	0.0546 (14)	0.0049 (12)	0.0032 (12)	0.0053 (11)
C(7)	0.0614 (19)	0.0715 (18)	0.0712 (17)	0.0147 (15)	0.0019 (14)	0.0040 (14)
C(8)	0.063 (2)	0.0660 (18)	0.109 (3)	0.0128 (15)	−0.0068 (18)	0.0108 (17)
C(9)	0.080 (3)	0.071 (2)	0.086 (3)	−0.0072 (17)	−0.0249 (18)	0.0174 (16)
C(10)	0.091 (3)	0.083 (2)	0.0624 (17)	−0.0093 (19)	−0.0075 (17)	−0.0003 (15)
C(11)	0.0663 (19)	0.0701 (17)	0.0639 (17)	0.0031 (14)	0.0038 (14)	−0.0034 (13)
C(12)	0.0423 (15)	0.0572 (14)	0.0580 (15)	−0.0024 (12)	0.0021 (12)	0.0057 (11)
C(13)	0.0521 (17)	0.0688 (17)	0.0670 (16)	0.0033 (14)	0.0018 (14)	0.0059 (13)
C(14)	0.077 (3)	0.0753 (19)	0.0592 (16)	0.0003 (16)	−0.0070 (15)	0.0029 (14)
C(15)	0.081 (3)	0.0638 (17)	0.0604 (16)	−0.0040 (16)	0.0129 (16)	0.0014 (13)
C(16)	0.0560 (18)	0.0653 (17)	0.089 (2)	0.0075 (14)	0.0120 (16)	−0.0044 (15)
C(17)	0.0489 (17)	0.0655 (16)	0.0741 (17)	0.0055 (13)	−0.0023 (13)	−0.0065 (13)
C(18)	0.0468 (16)	0.0497 (14)	0.0682 (16)	0.0050 (12)	0.0050 (13)	0.0004 (12)
C(19)	0.0449 (16)	0.0562 (14)	0.0567 (14)	−0.0011 (12)	0.0094 (12)	−0.0019 (11)
C(20)	0.0558 (18)	0.090 (2)	0.0554 (16)	0.0107 (16)	0.0102 (14)	0.0036 (14)
C(21)	0.073 (2)	0.132 (3)	0.0592 (17)	0.015 (2)	0.0067 (15)	0.0116 (17)
C(22)	0.093 (3)	0.146 (4)	0.0635 (19)	0.006 (3)	0.0223 (19)	−0.0039 (18)
C(23)	0.069 (2)	0.107 (3)	0.0762 (19)	0.0100 (18)	0.0269 (17)	−0.0053 (17)

Geometric parameters (Å, º)

Cl(1)—C(15)	1.745 (3)	C(20)—C(21)	1.497 (4)
O(1)—C(2)	1.418 (3)	C(21)—C(22)	1.485 (5)
O(1)—C(5)	1.434 (3)	C(22)—C(23)	1.505 (4)
O(2)—C(18)	1.221 (3)	C(2)—H(2)	0.980
C(2)—C(3)	1.573 (4)	C(3)—H(3)	0.980
C(2)—C(12)	1.509 (4)	C(4)—H(4A)	0.970
C(3)—C(4)	1.526 (4)	C(4)—H(4B)	0.970
C(3)—C(18)	1.511 (4)	C(5)—H(5)	0.980
C(4)—C(5)	1.521 (4)	C(7)—H(7)	0.930
C(5)—C(6)	1.491 (4)	C(8)—H(8)	0.930
C(6)—C(7)	1.376 (4)	C(9)—H(9)	0.930
C(6)—C(11)	1.381 (4)	C(10)—H(10)	0.930
C(7)—C(8)	1.382 (4)	C(11)—H(11)	0.930
C(8)—C(9)	1.366 (5)	C(13)—H(13)	0.930
C(9)—C(10)	1.363 (5)	C(14)—H(14)	0.930
C(10)—C(11)	1.382 (4)	C(16)—H(16)	0.930
C(12)—C(13)	1.382 (4)	C(17)—H(17)	0.930
C(12)—C(17)	1.386 (4)	C(20)—H(20)	0.96 (3)
C(13)—C(14)	1.379 (4)	C(21)—H(21A)	0.970
C(14)—C(15)	1.370 (5)	C(21)—H(21B)	0.970
C(15)—C(16)	1.375 (4)	C(22)—H(22A)	0.970
C(16)—C(17)	1.380 (4)	C(22)—H(22B)	0.970
C(18)—C(19)	1.467 (4)	C(23)—H(23A)	0.970
C(19)—C(20)	1.318 (4)	C(23)—H(23B)	0.970
C(19)—C(23)	1.488 (4)		

O(2)···C(2)^{i}	3.460 (3)	H(8)···H(4A)^{iv}	3.5198
O(2)···C(8)^{ii}	3.575 (4)	H(8)···H(4B)^{iv}	3.3875
O(2)···C(13)^{i}	3.537 (4)	H(8)···H(5)^{i}	2.7276
C(2)···O(2)^{iii}	3.460 (3)	H(8)···H(11)^{i}	3.0564
C(8)···O(2)^{iv}	3.575 (4)	H(8)···H(23B)^{iv}	3.5412
C(8)···C(18)^{iv}	3.475 (4)	H(8)···H(20)^{iv}	3.2315
C(8)···C(19)^{iv}	3.572 (4)	H(9)···Cl(1)^{ii}	3.4433
C(11)···C(15)^{v}	3.582 (4)	H(9)···Cl(1)^{x}	3.5629
C(13)···O(2)^{iii}	3.537 (4)	H(9)···C(19)^{iv}	3.4151
C(15)···C(11)^{vi}	3.582 (4)	H(9)···C(20)^{iv}	3.4699
C(18)···C(8)^{ii}	3.475 (4)	H(9)···C(21)^{iv}	3.5022
C(19)···C(8)^{ii}	3.572 (4)	H(9)···C(22)^{iv}	3.5828
Cl(1)···H(9)^{iv}	3.4433	H(9)···C(23)^{iv}	3.4077
Cl(1)···H(9)^{vii}	3.5629	H(9)···H(11)^{i}	3.1235
Cl(1)···H(10)^{vi}	3.4222	H(9)···H(16)^{ii}	3.4677
Cl(1)···H(10)^{vii}	3.3528	H(9)···H(21B)^{iv}	3.0383
Cl(1)···H(11)^{vi}	3.3964	H(9)···H(22A)^{iv}	3.3010
Cl(1)···H(21B)^{viii}	3.5133	H(9)···H(23B)^{iv}	2.8899
Cl(1)···H(22A)^{ix}	3.1389	H(10)···Cl(1)^{v}	3.4222
O(1)···H(4B)^{vi}	2.7496	H(10)···Cl(1)^{x}	3.3528

O(1)···H(7)^{iii}	3.4997	H(10)···C(15)^{v}	3.5757
O(1)···H(8)^{iii}	3.3806	H(10)···H(14)^{v}	3.4412
O(2)···H(2)^{i}	2.6806	H(10)···H(16)^{ii}	3.0881
O(2)···H(3)^{i}	3.0662	H(10)···H(21B)^{vi}	2.8666
O(2)···H(8)^{ii}	3.2230	H(11)···Cl(1)^{v}	3.3964
O(2)···H(13)^{i}	2.6883	H(11)···C(8)^{iii}	3.2748
O(2)···H(20)^{i}	3.03 (3)	H(11)···C(9)^{iii}	3.3137
C(2)···H(4B)^{vi}	3.3645	H(11)···C(14)^{v}	3.2922
C(3)···H(8)^{ii}	3.3504	H(11)···C(15)^{v}	2.8298
C(5)···H(8)^{iii}	3.2463	H(11)···C(16)^{v}	2.8939
C(6)···H(8)^{iii}	3.3292	H(11)···C(17)^{v}	3.4096
C(6)···H(20)^{vi}	3.52 (3)	H(11)···H(8)^{iii}	3.0564
C(7)···H(2)^{i}	3.5722	H(11)···H(9)^{iii}	3.1235
C(7)···H(5)^{i}	3.1280	H(11)···H(16)^{v}	3.1385
C(7)···H(20)^{vi}	3.44 (3)	H(13)···O(2)^{iii}	2.6883
C(8)···H(5)^{i}	2.9455	H(13)···C(16)^{iii}	3.2505
C(8)···H(11)^{i}	3.2748	H(13)···C(17)^{iii}	3.2991
C(8)···H(20)^{vi}	3.29 (3)	H(13)···H(16)^{iii}	3.0320
C(9)···H(11)^{i}	3.3137	H(13)···H(17)^{iii}	3.1180
C(9)···H(16)^{ii}	3.2006	H(13)···H(23A)^{iii}	3.0864
C(9)···H(23B)^{iv}	3.2533	H(13)···H(23B)^{iii}	3.5534
C(9)···H(20)^{vi}	3.20 (3)	H(14)···C(21)^{viii}	3.0892
C(10)···H(16)^{ii}	2.9601	H(14)···C(22)^{viii}	3.1317
C(10)···H(21B)^{vi}	3.1472	H(14)···H(10)^{vi}	3.4412
C(10)···H(20)^{vi}	3.26 (3)	H(14)···H(16)^{iii}	3.0912
C(11)···H(8)^{iii}	3.2826	H(14)···H(21A)^{viii}	2.8734
C(11)···H(16)^{ii}	3.3447	H(14)···H(21B)^{viii}	2.8730
C(11)···H(20)^{vi}	3.41 (3)	H(14)···H(22A)^{viii}	2.7927
C(12)···H(4B)^{vi}	3.3030	H(14)···H(22B)^{viii}	3.0585
C(13)···H(16)^{iii}	3.2583	H(16)···C(9)^{iv}	3.2006
C(14)···H(11)^{vi}	3.2922	H(16)···C(10)^{iv}	2.9601
C(14)···H(16)^{iii}	3.2907	H(16)···C(11)^{iv}	3.3447
C(14)···H(21A)^{viii}	3.4806	H(16)···C(13)^{i}	3.2583
C(15)···H(10)^{vi}	3.5757	H(16)···C(14)^{i}	3.2907
C(15)···H(11)^{vi}	2.8298	H(16)···H(9)^{iv}	3.4677
C(15)···H(22A)^{ix}	3.5571	H(16)···H(10)^{iv}	3.0881
C(16)···H(11)^{vi}	2.8939	H(16)···H(11)^{vi}	3.1385
C(16)···H(13)^{i}	3.2505	H(16)···H(13)^{i}	3.0320
C(17)···H(4B)^{vi}	3.3692	H(16)···H(14)^{i}	3.0912
C(17)···H(11)^{vi}	3.4096	H(17)···H(2)^{i}	3.2711
C(17)···H(13)^{i}	3.2991	H(17)···H(4B)^{vi}	3.2998
C(18)···H(8)^{ii}	2.8538	H(17)···H(13)^{i}	3.1180
C(19)···H(8)^{ii}	2.9837	H(21A)···C(14)^{viii}	3.4806
C(19)···H(9)^{ii}	3.4151	H(21A)···C(23)^{iii}	3.1440
C(20)···H(8)^{ii}	3.2142	H(21A)···H(14)^{viii}	2.8734
C(20)···H(9)^{ii}	3.4699	H(21A)···H(22A)^{iii}	3.3502
C(20)···H(23B)^{iii}	3.1048	H(21A)···H(22B)^{viii}	2.7457
C(21)···H(9)^{ii}	3.5022	H(21A)···H(23A)^{iii}	3.2912

C(21)···H(14)^{viii}	3.0892	H(21A)···H(23B)^{iii}	2.3173
C(21)···H(22B)^{viii}	3.5726	H(21B)···Cl(1)^{viii}	3.5133
C(21)···H(23B)^{iii}	3.0209	H(21B)···C(10)^{v}	3.1472
C(22)···H(9)^{ii}	3.5828	H(21B)···H(9)^{ii}	3.0383
C(22)···H(14)^{viii}	3.1317	H(21B)···H(10)^{v}	2.8666
C(22)···H(22B)^{ix}	3.4041	H(21B)···H(14)^{viii}	2.8730
C(22)···H(23A)^{ix}	3.1476	H(21B)···H(23B)^{iii}	3.4145
C(23)···H(9)^{ii}	3.4077	H(22A)···Cl(1)^{ix}	3.1389
C(23)···H(21A)^{i}	3.1440	H(22A)···C(15)^{ix}	3.5571
C(23)···H(22B)^{ix}	3.1328	H(22A)···H(9)^{ii}	3.3010
H(2)···O(2)^{iii}	2.6806	H(22A)···H(14)^{viii}	2.7927
H(2)···C(7)^{iii}	3.5722	H(22A)···H(21A)^{i}	3.3502
H(2)···H(4A)^{iii}	3.2970	H(22A)···H(22B)^{ix}	3.3354
H(2)···H(4B)^{vi}	3.4065	H(22A)···H(23A)^{ix}	2.8646
H(2)···H(7)^{iii}	2.8478	H(22B)···C(21)^{viii}	3.5726
H(2)···H(17)^{iii}	3.2711	H(22B)···C(22)^{ix}	3.4041
H(3)···O(2)^{iii}	3.0662	H(22B)···C(23)^{ix}	3.1328
H(3)···H(8)^{ii}	3.1877	H(22B)···H(14)^{viii}	3.0585
H(4A)···H(2)^{i}	3.2970	H(22B)···H(21A)^{viii}	2.7457
H(4A)···H(7)^{ii}	3.4183	H(22B)···H(22A)^{ix}	3.3354
H(4A)···H(8)^{ii}	3.5198	H(22B)···H(22B)^{viii}	3.0914
H(4B)···O(1)^{v}	2.7496	H(22B)···H(22B)^{ix}	3.1672
H(4B)···C(2)^{v}	3.3645	H(22B)···H(23A)^{ix}	2.6197
H(4B)···C(12)^{v}	3.3030	H(22B)···H(23B)^{ix}	3.0847
H(4B)···C(17)^{v}	3.3692	H(23A)···C(22)^{ix}	3.1476
H(4B)···H(2)^{v}	3.4065	H(23A)···H(13)^{i}	3.0864
H(4B)···H(7)^{ii}	3.4863	H(23A)···H(21A)^{i}	3.2912
H(4B)···H(8)^{ii}	3.3875	H(23A)···H(22A)^{ix}	2.8646
H(4B)···H(17)^{v}	3.2998	H(23A)···H(22B)^{ix}	2.6197
H(5)···C(7)^{iii}	3.1280	H(23B)···C(9)^{ii}	3.2533
H(5)···C(8)^{iii}	2.9455	H(23B)···C(20)^{i}	3.1048
H(5)···H(7)^{iii}	3.0467	H(23B)···C(21)^{i}	3.0209
H(5)···H(8)^{iii}	2.7276	H(23B)···H(8)^{ii}	3.5412
H(7)···O(1)^{i}	3.4997	H(23B)···H(9)^{ii}	2.8899
H(7)···H(2)^{i}	2.8478	H(23B)···H(13)^{i}	3.5534
H(7)···H(4A)^{iv}	3.4183	H(23B)···H(21A)^{i}	2.3173
H(7)···H(4B)^{iv}	3.4863	H(23B)···H(21B)^{i}	3.4145
H(7)···H(5)^{i}	3.0467	H(23B)···H(22B)^{ix}	3.0847
H(8)···O(1)^{i}	3.3806	H(23B)···H(20)^{i}	2.8072
H(8)···O(2)^{iv}	3.2230	H(20)···O(2)^{iii}	3.03 (3)
H(8)···C(3)^{iv}	3.3504	H(20)···C(6)^{v}	3.52 (3)
H(8)···C(5)^{i}	3.2463	H(20)···C(7)^{v}	3.44 (3)
H(8)···C(6)^{i}	3.3292	H(20)···C(8)^{v}	3.29 (3)
H(8)···C(11)^{i}	3.2826	H(20)···C(9)^{v}	3.20 (3)
H(8)···C(18)^{iv}	2.8538	H(20)···C(10)^{v}	3.26 (3)
H(8)···C(19)^{iv}	2.9837	H(20)···C(11)^{v}	3.41 (3)
H(8)···C(20)^{iv}	3.2142	H(20)···H(8)^{ii}	3.2315
H(8)···H(3)^{iv}	3.1877	H(20)···H(23B)^{iii}	2.8072

C(2)—O(1)—C(5)	107.08 (17)	C(18)—C(3)—H(3)	109.756
O(1)—C(2)—C(3)	105.33 (18)	C(3)—C(4)—H(4A)	110.822
O(1)—C(2)—C(12)	109.62 (19)	C(3)—C(4)—H(4B)	110.830
C(3)—C(2)—C(12)	116.0 (2)	C(5)—C(4)—H(4A)	110.820
C(2)—C(3)—C(4)	103.30 (19)	C(5)—C(4)—H(4B)	110.820
C(2)—C(3)—C(18)	110.98 (19)	H(4A)—C(4)—H(4B)	108.881
C(4)—C(3)—C(18)	113.2 (2)	O(1)—C(5)—H(5)	109.307
C(3)—C(4)—C(5)	104.6 (2)	C(4)—C(5)—H(5)	109.305
O(1)—C(5)—C(4)	103.60 (18)	C(6)—C(5)—H(5)	109.316
O(1)—C(5)—C(6)	110.1 (2)	C(6)—C(7)—H(7)	119.692
C(4)—C(5)—C(6)	115.0 (2)	C(8)—C(7)—H(7)	119.701
C(5)—C(6)—C(7)	121.2 (3)	C(7)—C(8)—H(8)	119.783
C(5)—C(6)—C(11)	120.5 (3)	C(9)—C(8)—H(8)	119.795
C(7)—C(6)—C(11)	118.2 (3)	C(8)—C(9)—H(9)	120.091
C(6)—C(7)—C(8)	120.6 (3)	C(10)—C(9)—H(9)	120.106
C(7)—C(8)—C(9)	120.4 (3)	C(9)—C(10)—H(10)	120.016
C(8)—C(9)—C(10)	119.8 (3)	C(11)—C(10)—H(10)	120.013
C(9)—C(10)—C(11)	120.0 (3)	C(6)—C(11)—H(11)	119.505
C(6)—C(11)—C(10)	121.0 (3)	C(10)—C(11)—H(11)	119.501
C(2)—C(12)—C(13)	119.8 (3)	C(12)—C(13)—H(13)	119.632
C(2)—C(12)—C(17)	121.6 (2)	C(14)—C(13)—H(13)	119.645
C(13)—C(12)—C(17)	118.5 (3)	C(13)—C(14)—H(14)	120.183
C(12)—C(13)—C(14)	120.7 (3)	C(15)—C(14)—H(14)	120.201
C(13)—C(14)—C(15)	119.6 (3)	C(15)—C(16)—H(16)	120.539
Cl(1)—C(15)—C(14)	119.9 (2)	C(17)—C(16)—H(16)	120.562
Cl(1)—C(15)—C(16)	119.0 (3)	C(12)—C(17)—H(17)	119.414
C(14)—C(15)—C(16)	121.1 (3)	C(16)—C(17)—H(17)	119.421
C(15)—C(16)—C(17)	118.9 (3)	C(19)—C(20)—H(20)	124.5 (15)
C(12)—C(17)—C(16)	121.2 (3)	C(21)—C(20)—H(20)	123.6 (15)
O(2)—C(18)—C(3)	121.0 (2)	C(20)—C(21)—H(21A)	110.941
O(2)—C(18)—C(19)	119.8 (3)	C(20)—C(21)—H(21B)	110.944
C(3)—C(18)—C(19)	119.2 (3)	C(22)—C(21)—H(21A)	110.955
C(18)—C(19)—C(20)	127.7 (3)	C(22)—C(21)—H(21B)	110.945
C(18)—C(19)—C(23)	120.9 (3)	H(21A)—C(21)—H(21B)	108.957
C(20)—C(19)—C(23)	111.3 (3)	C(21)—C(22)—H(22A)	110.164
C(19)—C(20)—C(21)	111.8 (3)	C(21)—C(22)—H(22B)	110.161
C(20)—C(21)—C(22)	104.1 (3)	C(23)—C(22)—H(22A)	110.158
C(21)—C(22)—C(23)	107.7 (3)	C(23)—C(22)—H(22B)	110.167
C(19)—C(23)—C(22)	104.2 (3)	H(22A)—C(22)—H(22B)	108.463
O(1)—C(2)—H(2)	108.562	C(19)—C(23)—H(23A)	110.912
C(3)—C(2)—H(2)	108.556	C(19)—C(23)—H(23B)	110.920
C(12)—C(2)—H(2)	108.535	C(22)—C(23)—H(23A)	110.929
C(2)—C(3)—H(3)	109.737	C(22)—C(23)—H(23B)	110.927
C(4)—C(3)—H(3)	109.735	H(23A)—C(23)—H(23B)	108.947

C(2)—O(1)—C(5)—C(4)	−41.7 (2)	C(11)—C(6)—C(7)—C(8)	−0.6 (4)
C(2)—O(1)—C(5)—C(6)	−165.24 (17)	C(6)—C(7)—C(8)—C(9)	−0.0 (4)

C(5)—O(1)—C(2)—C(3)	34.0 (2)	C(7)—C(8)—C(9)—C(10)	0.4 (5)
C(5)—O(1)—C(2)—C(12)	159.47 (17)	C(8)—C(9)—C(10)—C(11)	−0.1 (5)
O(1)—C(2)—C(3)—C(4)	−12.5 (3)	C(9)—C(10)—C(11)—C(6)	−0.6 (5)
O(1)—C(2)—C(3)—C(18)	109.02 (18)	C(2)—C(12)—C(13)—C(14)	175.9 (2)
O(1)—C(2)—C(12)—C(13)	151.51 (18)	C(2)—C(12)—C(17)—C(16)	−175.4 (2)
O(1)—C(2)—C(12)—C(17)	−31.7 (3)	C(13)—C(12)—C(17)—C(16)	1.4 (4)
C(3)—C(2)—C(12)—C(13)	−89.4 (3)	C(17)—C(12)—C(13)—C(14)	−1.0 (4)
C(3)—C(2)—C(12)—C(17)	87.4 (3)	C(12)—C(13)—C(14)—C(15)	−0.2 (4)
C(12)—C(2)—C(3)—C(4)	−133.95 (19)	C(13)—C(14)—C(15)—Cl(1)	179.5 (2)
C(12)—C(2)—C(3)—C(18)	−12.4 (3)	C(13)—C(14)—C(15)—C(16)	1.1 (4)
C(2)—C(3)—C(4)—C(5)	−11.7 (3)	Cl(1)—C(15)—C(16)—C(17)	−179.07 (17)
C(2)—C(3)—C(18)—O(2)	−85.7 (3)	C(14)—C(15)—C(16)—C(17)	−0.6 (4)
C(2)—C(3)—C(18)—C(19)	91.9 (3)	C(15)—C(16)—C(17)—C(12)	−0.6 (4)
C(4)—C(3)—C(18)—O(2)	29.9 (3)	O(2)—C(18)—C(19)—C(20)	−162.7 (2)
C(4)—C(3)—C(18)—C(19)	−152.55 (18)	O(2)—C(18)—C(19)—C(23)	13.7 (4)
C(18)—C(3)—C(4)—C(5)	−131.75 (19)	C(3)—C(18)—C(19)—C(20)	19.7 (4)
C(3)—C(4)—C(5)—O(1)	31.9 (3)	C(3)—C(18)—C(19)—C(23)	−163.86 (18)
C(3)—C(4)—C(5)—C(6)	152.05 (19)	C(18)—C(19)—C(20)—C(21)	176.2 (2)
O(1)—C(5)—C(6)—C(7)	42.3 (3)	C(18)—C(19)—C(23)—C(22)	177.59 (19)
O(1)—C(5)—C(6)—C(11)	−140.95 (19)	C(20)—C(19)—C(23)—C(22)	−5.5 (3)
C(4)—C(5)—C(6)—C(7)	−74.2 (3)	C(23)—C(19)—C(20)—C(21)	−0.5 (3)
C(4)—C(5)—C(6)—C(11)	102.5 (3)	C(19)—C(20)—C(21)—C(22)	6.3 (4)
C(5)—C(6)—C(7)—C(8)	176.2 (2)	C(20)—C(21)—C(22)—C(23)	−9.4 (4)
C(5)—C(6)—C(11)—C(10)	−175.9 (2)	C(21)—C(22)—C(23)—C(19)	9.2 (4)
C(7)—C(6)—C(11)—C(10)	0.9 (4)		

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+3/2, *y*+1/2, −*z*+3/2; (iii) *x*−1, *y*, *z*; (iv) −*x*+3/2, *y*−1/2, −*z*+3/2; (v) −*x*+1/2, *y*+1/2, −*z*+3/2; (vi) −*x*+1/2, *y*−1/2, −*z*+3/2; (vii) *x*−1/2, −*y*+1/2, *z*−1/2; (viii) −*x*, −*y*+1, −*z*+1; (ix) −*x*+1, −*y*+1, −*z*+1; (x) *x*+1/2, −*y*+1/2, *z*+1/2.