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Ethyl 2,6-bis(4-chlorophenyl)-1-isocyano-4-oxocyclohexanecarboxylate

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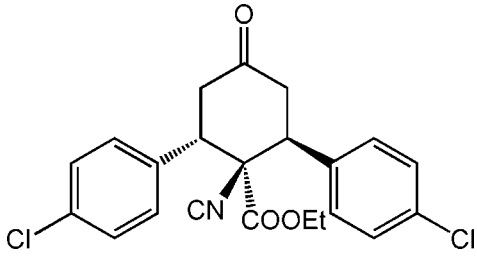
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.117; data-to-parameter ratio = 14.2.

In the title compound, $C_{22}H_{19}Cl_2NO_3$, the central six-membered ring is in a twist-boat conformation. The two aryl groups are in equatorial positions, *trans* to each other and with a dihedral angle of 77.50 (2)° between them. One of the least hindered $-CH_2-$ groups and one of the aryl-substituted C atoms, with its axial H atom, are in the flagpole positions. The ethoxycarbonyl group is in an equatorial position and is *cis* to the second aryl group. In the crystal, molecules are linked *via* weak C—H···O hydrogen bonds, forming chains along [010].

Related literature

For the synthesis, see: Zhang *et al.* (2010); Tan *et al.* (2009). For related structures, see: Rowland & Gill (1988); Aleman *et al.* (2009); Wu *et al.* (2011); Li *et al.* (2011). For other [5 + 1] annulation reactions, see: Bi *et al.* (2005); Zhao *et al.* (2006); Fu *et al.* (2009); Xu *et al.* (2012).



Experimental

Crystal data

$C_{22}H_{19}Cl_2NO_3$
M_r = 416.28
Monoclinic, $C2/c$
a = 21.6980 (17) Å
b = 11.0770 (19) Å
c = 17.515 (3) Å
β = 104.535 (2)°
V = 4075.0 (10) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 293 K
0.21 × 0.19 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.932, T_{max} = 0.951
9983 measured reflections
3602 independent reflections
2584 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.117
S = 1.01
3602 reflections
253 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···O1^{i}	0.98	2.57	3.218 (3)	123

Symmetry code: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: LR2127).

References

Aleman, C., Jiménez, A. I., Cativiela, C., Nussinov, R. & Casanovas, J. (2009). *J. Org. Chem.* **74**, 7834–7843.
Bi, X., Dong, D., Liu, Q., Pan, W., Zhao, L. & Li, B. (2005). *J. Am. Chem. Soc.* **127**, 4578–4579.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fu, Z., Wang, M., Dong, Y., Liu, J. & Liu, Q. (2009). *J. Org. Chem.* **74**, 6105–6110.
Li, Y., Xu, X., Tan, J., Xia, C., Zhang, D. & Liu, Q. (2011). *J. Am. Chem. Soc.* **133**, 1775–1777.
Rowland, A. T. & Gill, B. C. (1988). *J. Org. Chem.* **53**, 434–437.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tan, J., Xu, X., Zhang, L., Li, Y. & Liu, Q. (2009). *Angew. Chem. Int. Ed.* **48**, 2868–2872.
Wu, B., Liu, G., Li, M., Zhang, Y., Zhang, S., Qiu, J., Xu, X., Ji, S. & Wang, X. (2011). *Chem. Commun.* **47**, 3992–3994.
Xu, X., Liu, Y. & Park, C. (2012). *Angew. Chem. Int. Ed.* **51**, 9372–9376.
Zhang, D., Xu, X. & &Liu, Q. (2010). *Synlett*, **6**, 917–920.
Zhao, L., Liang, F., Bi, X., Sun, S. & Liu, Q. (2006). *J. Org. Chem.* **71**, 1094–1098.

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Ethyl 2,6-bis(4-chlorophenyl)-1-isocyano-4-oxocyclohexanecarboxylate

Dawei Zhang, Peng Yang, Wei Liu and Jing Li

S1. Experimental

S1.1. Synthesis and crystallization

To the mixture of 1,5-bis(4-chlorophenyl)penta-1,4-dien-3-one (303 mg, 1.0 mmol) and ethyl isocyanoacetate (0.132 mL, 1.2 mmol) in DMF (5 mL) was added 1,8-diazabicyclo [5.4.0]undec-7-ene (DBU) (0.015 mL, 0.1 mmol) in one portion at room temperature. The reaction mixture was stirred at room temperature, and the reaction mixture was monitored by TLC. After the substrate 1,5-bis(4-chlorophenyl)penta-1,4-dien-3-one was consumed, the resulting mixture was poured into ice-water (30 mL) under stirring. The precipitated solid was collected by filtration, washed with water (3 × 10 mL), and dried under vacuum to afford the crude product, which was purified by flash chromatography (silica gel, petroleum ether : diethyl ether = 3:1, V/V) to give ethyl 2,6-bis(4-chlorophenyl)-1-isocyano-4-oxocyclohexanecarboxylate (387 mg, 93%). The material was recrystallized from a mixture of petroleum ether and diethyl ether to provide a crystalline solid.

S1.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.Hydrogen atoms were generated in idealized positions (according to the *sp*2 or *sp*3 geometries of their parent carbon), and then refined using a riding model with fixed C—H distances (C—H = 0.95–1.00 Å) and with *U*iso(H) = 1.2*U*eq(C).

S2. Results and discussion

[5+1] annulation is a novel strategy for the construction of six-membered cyclic compounds and total synthesis of natural products (Rowland & Gill, 1988; Wu *et al.*, 2011; Li *et al.*, 2011). The regiospecific [5+1] annulation reactions have drawn much attentions and both the five-carbon 1,5-bielectrophiles and the one-atom nucleophiles been explored extensively (Bi *et al.*, 2005; Zhao *et al.*, 2006; Fu *et al.*, 2009; Xu *et al.*, 2012). We have been dealing with functionalized ketene dithioacetals for several years and have succeeded in the preparation of six-membered aromatic and heterocyclic compounds based on [5C+1X] annulations (Zhang *et al.*, 2010; Tan *et al.*, 2009). The aromatic cyclic compounds are analogues of phenylalanine (Phe) which are potential moieties for the synthesis of peptide analogues with controlled fold in the backbone. The constrained ring systems play important roles in restricting torsional angle χ1 and in peptide receptor recognition processes (Aleman *et al.*, 2009).

The crystal structure of title compound, a phenyl substituted highly constrained cyclohexane analogue of Ph, is reported in this paper. Due to the steric hindrance, the oxocyclohexane is in a twist-boat conformation (Fig. 1). The ethoxyl carbonyl and the two aryl groups are located in equatorial positions. The dihedral angle between two aromatic rings is 77.495 (20)°. The C7 axial hydrogen and the CH_2 bonded to C10 are on the flagpole positions of the boat conformation, which give the least torsional strain. The equatorial ethoxyl carbonyl on C18 and the equatorial aryl group on C11 also lead the formation of a comparable stable boat conformation of this compound.

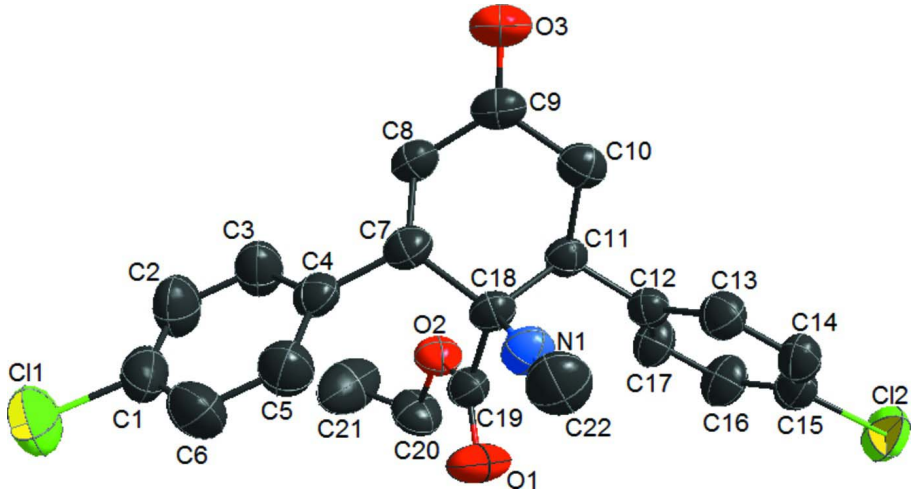

Figure 1

View of the molecular structure of the title compound with labeling and displacement ellipsoids drawn at the 30% probability level.

Ethyl 2,6-bis(4-chlorophenyl)-1-isocyano-4-oxocyclohexanecarboxylate

Crystal data

$C_{22}H_{19}Cl_2NO_3$
$M_r = 416.28$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 21.6980\ (17)$ Å
$b = 11.0770\ (19)$ Å
$c = 17.515\ (3)$ Å
$\beta = 104.535\ (2)°$
$V = 4075.0\ (10)$ Å^3
$Z = 8$
$F(000) = 1728$
$D_x = 1.357$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 106 reflections
$\theta = 1.3–26.0°$
$\mu = 0.34$ mm^{-1}
$T = 293$ K
BLOCK, colorless
0.21 × 0.19 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.932$, $T_{max} = 0.951$
9983 measured reflections
3602 independent reflections
2584 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.9°$
$h = -25\rightarrow25$
$k = -13\rightarrow13$
$l = -20\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.117$
$S = 1.01$
3602 reflections
253 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-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 2.7869P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
Cl2	−0.01505 (3)	0.07074 (6)	0.62309 (5)	0.0780 (2)
Cl1	0.59500 (4)	0.06816 (9)	0.99930 (6)	0.1128 (4)
O2	0.30900 (7)	0.21848 (13)	0.84990 (9)	0.0528 (4)
C12	0.18929 (9)	0.19357 (17)	0.66846 (12)	0.0443 (5)
C18	0.30971 (9)	0.15687 (17)	0.72324 (12)	0.0435 (5)
N1	0.30407 (9)	0.04712 (16)	0.67773 (12)	0.0524 (5)
C11	0.25594 (9)	0.24575 (18)	0.68262 (12)	0.0424 (5)
H11	0.2578	0.3129	0.7196	0.051*
C4	0.42949 (9)	0.17517 (19)	0.79764 (14)	0.0492 (5)
O1	0.28944 (9)	0.02289 (15)	0.82275 (11)	0.0759 (5)
C7	0.37764 (9)	0.21213 (18)	0.72628 (13)	0.0477 (5)
H7	0.3901	0.1767	0.6811	0.057*
C19	0.30148 (9)	0.12206 (19)	0.80460 (13)	0.0475 (5)
C9	0.33050 (11)	0.3749 (2)	0.63092 (15)	0.0552 (6)
C17	0.15361 (10)	0.2176 (2)	0.72194 (14)	0.0561 (6)
H17	0.1719	0.2608	0.7676	0.067*
O3	0.34259 (9)	0.45029 (17)	0.58723 (12)	0.0832 (6)
C14	0.09922 (11)	0.0868 (2)	0.58857 (14)	0.0567 (6)
H14	0.0811	0.0413	0.5440	0.068*
C10	0.27117 (10)	0.2997 (2)	0.60953 (13)	0.0517 (5)
H10A	0.2767	0.2352	0.5744	0.062*
H10B	0.2358	0.3494	0.5818	0.062*
C15	0.06450 (10)	0.1152 (2)	0.64196 (15)	0.0551 (6)
C8	0.37399 (10)	0.34828 (19)	0.71039 (14)	0.0542 (6)
H8A	0.3584	0.3888	0.7509	0.065*
H8B	0.4162	0.3791	0.7125	0.065*
C13	0.16142 (10)	0.12688 (19)	0.60210 (13)	0.0526 (6)
H13	0.1849	0.1086	0.5659	0.063*
C20	0.30188 (13)	0.2027 (3)	0.93036 (14)	0.0705 (7)
H20A	0.2595	0.2272	0.9327	0.085*
H20B	0.3074	0.1183	0.9452	0.085*
C16	0.09146 (11)	0.1789 (2)	0.70886 (16)	0.0646 (7)

H16	0.0680	0.1961	0.7453	0.078*
C3	0.46190 (11)	0.2575 (2)	0.85150 (16)	0.0690 (7)
H3	0.4496	0.3381	0.8459	0.083*
C5	0.44867 (12)	0.0562 (2)	0.80921 (17)	0.0694 (7)
H5	0.4275	−0.0025	0.7744	0.083*
C1	0.53022 (11)	0.1084 (3)	0.92278 (17)	0.0717 (7)
C6	0.49881 (14)	0.0224 (3)	0.87177 (19)	0.0829 (9)
H6	0.5109	−0.0582	0.8789	0.099*
C22	0.30053 (14)	−0.0380 (2)	0.63980 (18)	0.0765 (8)
C2	0.51188 (12)	0.2252 (3)	0.91352 (18)	0.0793 (8)
H2	0.5329	0.2834	0.9489	0.095*
C21	0.34862 (18)	0.2745 (3)	0.98441 (18)	0.1104 (12)
H21A	0.3437	0.2640	1.0370	0.166*
H21B	0.3428	0.3581	0.9698	0.166*
H21C	0.3905	0.2493	0.9825	0.166*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2	0.0464 (3)	0.0811 (5)	0.1010 (6)	−0.0096 (3)	0.0083 (3)	−0.0033 (4)
Cl1	0.0699 (5)	0.1466 (8)	0.1075 (7)	0.0144 (5)	−0.0045 (4)	0.0360 (6)
O2	0.0637 (9)	0.0523 (9)	0.0461 (9)	−0.0020 (7)	0.0207 (7)	0.0016 (7)
C12	0.0474 (11)	0.0384 (10)	0.0454 (12)	0.0012 (9)	0.0087 (10)	0.0009 (9)
C18	0.0492 (11)	0.0345 (10)	0.0487 (13)	−0.0035 (8)	0.0159 (10)	−0.0030 (9)
N1	0.0580 (11)	0.0386 (10)	0.0609 (12)	−0.0003 (8)	0.0153 (9)	−0.0051 (9)
C11	0.0472 (11)	0.0377 (10)	0.0428 (12)	−0.0030 (8)	0.0122 (9)	−0.0017 (9)
C4	0.0417 (11)	0.0520 (12)	0.0586 (14)	−0.0016 (9)	0.0216 (10)	0.0030 (11)
O1	0.1060 (14)	0.0511 (10)	0.0770 (13)	−0.0162 (9)	0.0350 (11)	0.0125 (9)
C7	0.0480 (12)	0.0463 (12)	0.0534 (13)	−0.0034 (9)	0.0210 (10)	−0.0009 (10)
C19	0.0430 (11)	0.0458 (12)	0.0543 (14)	−0.0024 (9)	0.0136 (10)	0.0049 (11)
C9	0.0647 (14)	0.0492 (12)	0.0582 (15)	−0.0031 (11)	0.0275 (12)	0.0072 (12)
C17	0.0511 (13)	0.0594 (14)	0.0583 (15)	−0.0059 (10)	0.0147 (11)	−0.0144 (12)
O3	0.0888 (13)	0.0857 (13)	0.0778 (13)	−0.0170 (10)	0.0257 (11)	0.0318 (11)
C14	0.0580 (14)	0.0522 (13)	0.0528 (14)	−0.0055 (10)	0.0009 (11)	−0.0060 (11)
C10	0.0611 (13)	0.0484 (12)	0.0456 (13)	0.0006 (10)	0.0133 (11)	0.0013 (10)
C15	0.0456 (12)	0.0486 (12)	0.0674 (16)	−0.0023 (10)	0.0076 (11)	0.0037 (12)
C8	0.0538 (12)	0.0498 (13)	0.0609 (15)	−0.0114 (10)	0.0179 (11)	0.0043 (11)
C13	0.0548 (13)	0.0538 (13)	0.0491 (14)	−0.0020 (10)	0.0130 (11)	−0.0039 (11)
C20	0.0799 (17)	0.0865 (19)	0.0499 (15)	0.0054 (14)	0.0254 (14)	0.0099 (14)
C16	0.0535 (13)	0.0734 (16)	0.0714 (17)	−0.0050 (12)	0.0240 (13)	−0.0131 (14)
C3	0.0583 (14)	0.0608 (15)	0.0810 (19)	0.0014 (12)	0.0046 (14)	−0.0045 (14)
C5	0.0659 (15)	0.0577 (15)	0.0817 (19)	0.0030 (12)	0.0130 (14)	0.0013 (14)
C1	0.0472 (13)	0.095 (2)	0.0732 (18)	0.0015 (13)	0.0148 (13)	0.0141 (16)
C6	0.0760 (18)	0.0686 (17)	0.102 (2)	0.0186 (15)	0.0177 (17)	0.0211 (17)
C22	0.0902 (19)	0.0528 (15)	0.083 (2)	0.0022 (14)	0.0155 (16)	−0.0109 (15)
C2	0.0608 (16)	0.087 (2)	0.080 (2)	−0.0032 (14)	−0.0005 (15)	−0.0080 (16)
C21	0.143 (3)	0.130 (3)	0.0617 (19)	−0.051 (2)	0.033 (2)	−0.021 (2)

Geometric parameters (Å, º)

Cl2—C15	1.745 (2)	C14—C15	1.377 (3)
Cl1—C1	1.739 (3)	C14—C13	1.383 (3)
O2—C19	1.316 (3)	C14—H14	0.9300
O2—C20	1.466 (3)	C10—H10A	0.9700
C12—C13	1.382 (3)	C10—H10B	0.9700
C12—C17	1.383 (3)	C15—C16	1.367 (3)
C12—C11	1.519 (3)	C8—H8A	0.9700
C18—N1	1.442 (3)	C8—H8B	0.9700
C18—C19	1.529 (3)	C13—H13	0.9300
C18—C11	1.556 (3)	C20—C21	1.441 (4)
C18—C7	1.584 (3)	C20—H20A	0.9700
N1—C22	1.144 (3)	C20—H20B	0.9700
C11—C10	1.523 (3)	C16—H16	0.9300
C11—H11	0.9800	C3—C2	1.376 (4)
C4—C3	1.373 (3)	C3—H3	0.9300
C4—C5	1.382 (3)	C5—C6	1.388 (4)
C4—C7	1.513 (3)	C5—H5	0.9300
O1—C19	1.191 (3)	C1—C2	1.352 (4)
C7—C8	1.532 (3)	C1—C6	1.365 (4)
C7—H7	0.9800	C6—H6	0.9300
C9—O3	1.205 (3)	C2—H2	0.9300
C9—C10	1.500 (3)	C21—H21A	0.9600
C9—C8	1.502 (3)	C21—H21B	0.9600
C17—C16	1.378 (3)	C21—H21C	0.9600
C17—H17	0.9300		

C19—O2—C20	117.11 (18)	H10A—C10—H10B	108.0
C13—C12—C17	118.1 (2)	C16—C15—C14	120.7 (2)
C13—C12—C11	122.58 (19)	C16—C15—Cl2	119.88 (19)
C17—C12—C11	119.21 (19)	C14—C15—Cl2	119.39 (18)
N1—C18—C19	106.80 (16)	C9—C8—C7	110.72 (19)
N1—C18—C11	109.33 (17)	C9—C8—H8A	109.5
C19—C18—C11	109.64 (16)	C7—C8—H8A	109.5
N1—C18—C7	107.07 (16)	C9—C8—H8B	109.5
C19—C18—C7	113.03 (17)	C7—C8—H8B	109.5
C11—C18—C7	110.82 (15)	H8A—C8—H8B	108.1
C22—N1—C18	177.6 (2)	C12—C13—C14	121.2 (2)
C12—C11—C10	114.26 (17)	C12—C13—H13	119.4
C12—C11—C18	114.07 (16)	C14—C13—H13	119.4
C10—C11—C18	109.69 (16)	C21—C20—O2	109.8 (2)
C12—C11—H11	106.0	C21—C20—H20A	109.7
C10—C11—H11	106.0	O2—C20—H20A	109.7
C18—C11—H11	106.0	C21—C20—H20B	109.7
C3—C4—C5	116.7 (2)	O2—C20—H20B	109.7
C3—C4—C7	122.3 (2)	H20A—C20—H20B	108.2
C5—C4—C7	120.9 (2)	C15—C16—C17	119.6 (2)

C4—C7—C8	114.22 (18)	C15—C16—H16	120.2
C4—C7—C18	114.57 (17)	C17—C16—H16	120.2
C8—C7—C18	111.65 (16)	C4—C3—C2	122.4 (3)
C4—C7—H7	105.1	C4—C3—H3	118.8
C8—C7—H7	105.1	C2—C3—H3	118.8
C18—C7—H7	105.1	C4—C5—C6	121.4 (3)
O1—C19—O2	126.2 (2)	C4—C5—H5	119.3
O1—C19—C18	124.5 (2)	C6—C5—H5	119.3
O2—C19—C18	109.36 (17)	C2—C1—C6	120.4 (3)
O3—C9—C10	122.4 (2)	C2—C1—Cl1	119.6 (2)
O3—C9—C8	122.6 (2)	C6—C1—Cl1	120.0 (2)
C10—C9—C8	114.99 (18)	C1—C6—C5	119.5 (3)
C16—C17—C12	121.2 (2)	C1—C6—H6	120.2
C16—C17—H17	119.4	C5—C6—H6	120.2
C12—C17—H17	119.4	C1—C2—C3	119.6 (3)
C15—C14—C13	119.1 (2)	C1—C2—H2	120.2
C15—C14—H14	120.4	C3—C2—H2	120.2
C13—C14—H14	120.4	C20—C21—H21A	109.5
C9—C10—C11	111.22 (18)	C20—C21—H21B	109.5
C9—C10—H10A	109.4	H21A—C21—H21B	109.5
C11—C10—H10A	109.4	C20—C21—H21C	109.5
C9—C10—H10B	109.4	H21A—C21—H21C	109.5
C11—C10—H10B	109.4	H21B—C21—H21C	109.5

C19—C18—N1—C22	−164 (6)	C7—C18—C19—O2	59.8 (2)
C11—C18—N1—C22	78 (6)	C13—C12—C17—C16	−1.5 (3)
C7—C18—N1—C22	−43 (6)	C11—C12—C17—C16	175.7 (2)
C13—C12—C11—C10	41.3 (3)	O3—C9—C10—C11	−160.1 (2)
C17—C12—C11—C10	−135.7 (2)	C8—C9—C10—C11	20.6 (3)
C13—C12—C11—C18	−86.0 (2)	C12—C11—C10—C9	166.34 (18)
C17—C12—C11—C18	96.9 (2)	C18—C11—C10—C9	−64.1 (2)
N1—C18—C11—C12	55.2 (2)	C13—C14—C15—C16	−2.0 (4)
C19—C18—C11—C12	−61.5 (2)	C13—C14—C15—Cl2	177.18 (17)
C7—C18—C11—C12	173.01 (17)	O3—C9—C8—C7	−139.3 (2)
N1—C18—C11—C10	−74.4 (2)	C10—C9—C8—C7	40.1 (3)
C19—C18—C11—C10	168.84 (17)	C4—C7—C8—C9	169.06 (18)
C7—C18—C11—C10	43.4 (2)	C18—C7—C8—C9	−58.9 (2)
C3—C4—C7—C8	11.8 (3)	C17—C12—C13—C14	1.1 (3)
C5—C4—C7—C8	−164.7 (2)	C11—C12—C13—C14	−176.0 (2)
C3—C4—C7—C18	−118.7 (2)	C15—C14—C13—C12	0.7 (3)
C5—C4—C7—C18	64.7 (3)	C19—O2—C20—C21	140.9 (3)
N1—C18—C7—C4	−93.2 (2)	C14—C15—C16—C17	1.6 (4)
C19—C18—C7—C4	24.1 (2)	Cl2—C15—C16—C17	−177.61 (19)
C11—C18—C7—C4	147.63 (18)	C12—C17—C16—C15	0.2 (4)
N1—C18—C7—C8	134.96 (19)	C5—C4—C3—C2	1.1 (4)
C19—C18—C7—C8	−107.7 (2)	C7—C4—C3—C2	−175.6 (2)
C11—C18—C7—C8	15.8 (2)	C3—C4—C5—C6	−0.8 (4)
C20—O2—C19—O1	−0.1 (3)	C7—C4—C5—C6	176.0 (2)

C20—O2—C19—C18	179.17 (17)	C2—C1—C6—C5	1.3 (4)
N1—C18—C19—O1	−3.5 (3)	Cl1—C1—C6—C5	−177.6 (2)
C11—C18—C19—O1	114.8 (2)	C4—C5—C6—C1	−0.4 (4)
C7—C18—C19—O1	−121.0 (2)	C6—C1—C2—C3	−1.0 (4)
N1—C18—C19—O2	177.26 (16)	Cl1—C1—C2—C3	178.0 (2)
C11—C18—C19—O2	−64.4 (2)	C4—C3—C2—C1	−0.2 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O1^{i}	0.98	2.57	3.218 (3)	123

Symmetry code: (i) −*x*+1/2, *y*+1/2, −*z*+3/2.