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rac-5-Chloromethyl-3-(3-chloro-2-methylphenyl)-2,2-diphenyloxazolidine

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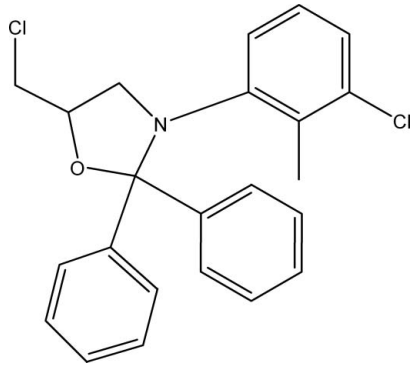
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.131; data-to-parameter ratio = 18.6.

In the title compound, $C_{23}H_{21}Cl_2NO$, the five-membered oxazolidine ring has a half-boat conformation, with a dihedral angle of 37.4 (2)° between the C_3O and C_2N planes.

Related literature

For related literature, see: Agami & Couty (2004).



Experimental

Crystal data

$C_{23}H_{21}Cl_2NO$
M_r = 398.31
Monoclinic, $C2/c$
a = 25.3638 (9) Å
b = 7.1591 (2) Å
c = 22.1688 (7) Å
β = 91.2630 (10)°
V = 4024.5 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 298 (1) K
0.53 × 0.48 × 0.39 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.834, T_{max} = 0.878
18483 measured reflections
4564 independent reflections
3844 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.131
S = 1.06
4564 reflections
245 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2169).

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supplementary materials

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rac-5-Chloromethyl-3-(3-chloro-2-methylphenyl)-2,2-diphenyloxazolidine

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Comment

5-Chloromethyl-3-(3-chloro-2-methyl-phenyl)-2,2-diphenyl-oxazolidine, (I), was widely used as ligand for metal-catalyzed asymmetric synthesis (Agami *et al.*, 2004). It was obtained from the reaction of 2-chloromethyl-oxirane and benzhydrylidene-(3-chloro-2-methyl- phenyl)-amine, as colorless crystals suitable for X-ray crystallographic analysis.

The molecular structure of (I) is built up from four rings, three of which are six-membered and one five-membered (Fig. 1). Atoms C1, C2, C3 and O1 are coplanar, the largest deviation being 0.0046 (10) Å for O1. Atom N1 deviates from the C1—C3/O1 plane by −0.5602 (23) Å. So the five membered oxazolidine ring has an half-boat conformation. The dihedral angles between the C1—C3/O1 plane and the C1/C3/N1 and C17—C22 planes are 37.42 (15)° and 89.86 (7)°, respectively. The dihedral angles between the C1—C3/O1 plane and the C5—C10 and C11—C16 planes are 69.24 (7)° and 26.39 (7)°, respectively. The molecule is chiral at C2 but as the space group is centrosymmetric, the unit cell contains the racemate (*R*,*S*).

Experimental

A mixture of 2-chloromethyl-oxirane (0.28 g, 3 mmol), benzhydrylidene-(3-chloro-2-methyl-phenyl)-amine (0.61 g, 2 mmol), and $Yb(OTf)_3$ (0.06 g, 5 mol%) was stirred at 40°C for 4 h. After completion of conversion as indicated by TLC, the reaction mixture was purified by silica gel column chromatography with petroleum ether-ethyl acetate (10:1) as eluent to afford the white solid (0.72 g, 91%). A solution of the compound in ethanol was concentrated gradually at room temperature to afford colourless chunks (m.p. 379–380 K).

Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic), 0.97 Å (methylene), 0.96 Å (CH_3) and 0.98 Å (methine) with $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(CH_3)$.

Figures

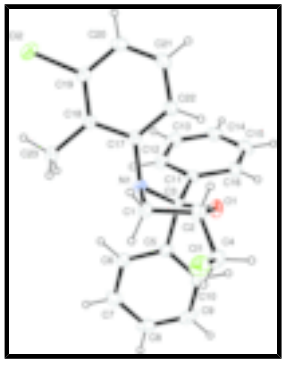


Fig. 1. The structure of (I), shown with 30% probability displacement ellipsoids.

rac-5-Chloromethyl-3-(3-chloro-2-methylphenyl)-2,2-diphenyloxazolidine

Crystal data

$C_{23}H_{21}Cl_2NO$	F_{000} = 1664
M_r = 398.31	D_x = 1.315 Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point: 380 K
Hall symbol: -C 2yc	Mo *K*α radiation λ = 0.71073 Å
a = 25.3638 (9) Å	Cell parameters from 14749 reflections
b = 7.1591 (2) Å	θ = 3.1–27.5º
c = 22.1688 (7) Å	μ = 0.34 mm^{-1}
β = 91.2630 (10)º	*T* = 298 (1) K
V = 4024.5 (2) Å^3	Chunk, colourless
Z = 8	0.53 × 0.48 × 0.39 mm

Data collection

Rigaku R-AXIS RAPID diffractometer	4564 independent reflections
Radiation source: fine-focus sealed tube	3844 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
Detector resolution: 10.00 pixels mm^{-1}	θ_{max} = 27.5º
T = 298(1) K	θ_{min} = 3.1º
ω scans	*h* = −32→32
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	*k* = −8→9
T_{min} = 0.834, T_{max} = 0.878	*l* = −28→28
18483 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.044	H-atom parameters constrained
$wR(F^2)$ = 0.131	$w = 1/[\sigma^2(F_o^2) + (0.0769P)^2 + 1.6308P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.046
4564 reflections	$\Delta\rho_{max}$ = 0.70 e Å^{-3}
245 parameters	$\Delta\rho_{min}$ = −0.27 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}
Cl1	−0.023418 (19)	0.76049 (8)	0.46113 (2)	0.06416 (17)
Cl2	0.06416 (2)	0.14708 (7)	0.14014 (2)	0.06141 (16)
O1	0.12341 (4)	0.69666 (16)	0.40447 (6)	0.0463 (3)
N1	0.10091 (5)	0.42695 (18)	0.35117 (5)	0.0360 (3)
C1	0.14307 (6)	0.5132 (2)	0.38770 (7)	0.0368 (3)
C2	0.05299 (6)	0.4908 (2)	0.38171 (8)	0.0445 (4)
H2A	0.0454	0.4120	0.4160	0.053*
H2B	0.0227	0.4905	0.3542	0.053*
C3	0.06683 (7)	0.6917 (3)	0.40219 (9)	0.0506 (4)
H3	0.0534	0.7815	0.3722	0.061*
C4	0.04747 (8)	0.7428 (3)	0.46360 (9)	0.0607 (5)
H4A	0.0627	0.8611	0.4762	0.073*
H4B	0.0583	0.6482	0.4927	0.073*
C5	0.15366 (6)	0.3955 (2)	0.44474 (7)	0.0395 (3)
C6	0.17638 (8)	0.4769 (3)	0.49598 (8)	0.0564 (5)
H6	0.1819	0.6053	0.4971	0.068*
C7	0.19084 (9)	0.3684 (4)	0.54548 (8)	0.0705 (6)
H7	0.2060	0.4247	0.5795	0.085*
C8	0.18294 (9)	0.1788 (4)	0.54477 (9)	0.0669 (6)
H8	0.1929	0.1064	0.5780	0.080*
C9	0.16013 (9)	0.0967 (3)	0.49440 (9)	0.0627 (5)
H9	0.1544	−0.0315	0.4937	0.075*
C10	0.14575 (7)	0.2039 (3)	0.44476 (8)	0.0498 (4)
H10	0.1306	0.1467	0.4110	0.060*
C11	0.19526 (6)	0.5360 (2)	0.35628 (6)	0.0382 (3)
C12	0.22422 (7)	0.7004 (3)	0.36172 (8)	0.0502 (4)
H12	0.2106	0.8018	0.3825	0.060*
C13	0.27367 (8)	0.7123 (3)	0.33595 (10)	0.0606 (5)
H13	0.2931	0.8220	0.3397	0.073*
C14	0.29399 (7)	0.5635 (3)	0.30504 (8)	0.0578 (5)
H14	0.3270	0.5728	0.2878	0.069*
C15	0.26552 (7)	0.4008 (3)	0.29954 (8)	0.0549 (5)
H15	0.2792	0.3001	0.2785	0.066*

C16	0.21637 (6)	0.3868 (3)	0.32534 (7)	0.0463 (4)
H16	0.1974	0.2760	0.3218	0.056*
C17	0.09890 (5)	0.4577 (2)	0.28690 (7)	0.0355 (3)
C18	0.11064 (7)	0.6283 (2)	0.26038 (8)	0.0474 (4)
H18	0.1202	0.7296	0.2845	0.057*
C19	0.10818 (8)	0.6480 (3)	0.19804 (9)	0.0551 (4)
H19	0.1164	0.7623	0.1806	0.066*
C20	0.09367 (7)	0.4997 (3)	0.16203 (8)	0.0502 (4)
H20	0.0921	0.5127	0.1203	0.060*
C21	0.08146 (6)	0.3310 (2)	0.18858 (7)	0.0415 (3)
C22	0.08338 (5)	0.3039 (2)	0.25121 (7)	0.0361 (3)
C23	0.07082 (8)	0.1201 (2)	0.28008 (8)	0.0482 (4)
H23A	0.0624	0.0298	0.2493	0.072*
H23B	0.0412	0.1349	0.3059	0.072*
H23C	0.1008	0.0778	0.3035	0.072*

Atomic displacement parameters (Å^2)

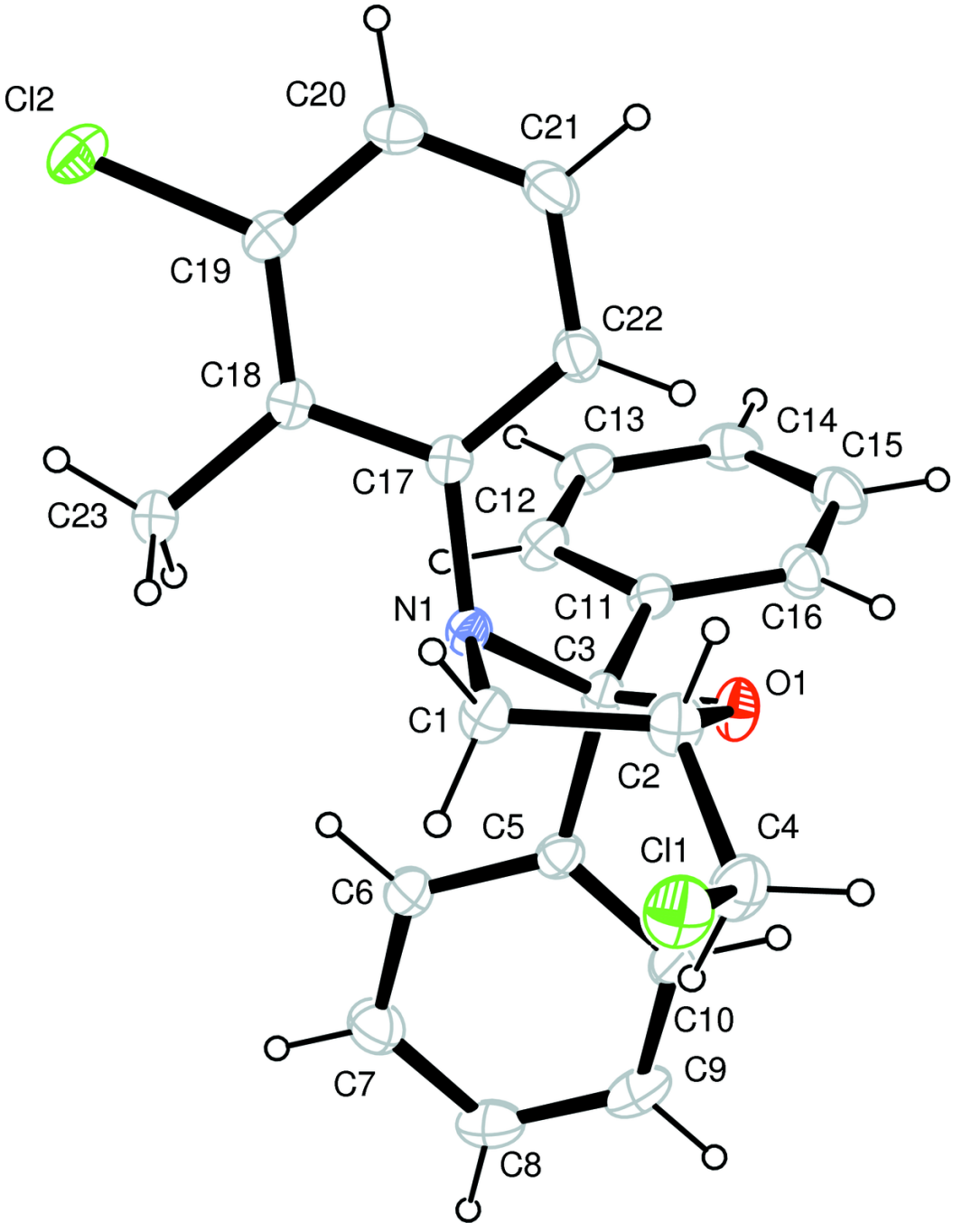
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0490 (3)	0.0713 (3)	0.0729 (3)	0.0029 (2)	0.0195 (2)	−0.0129 (2)
Cl2	0.0718 (3)	0.0654 (3)	0.0466 (2)	0.0037 (2)	−0.0071 (2)	−0.0167 (2)
O1	0.0391 (6)	0.0378 (6)	0.0623 (7)	−0.0018 (5)	0.0060 (5)	−0.0145 (5)
N1	0.0320 (6)	0.0386 (6)	0.0375 (6)	−0.0023 (5)	0.0014 (5)	−0.0036 (5)
C1	0.0355 (7)	0.0349 (7)	0.0401 (7)	−0.0033 (6)	0.0023 (6)	−0.0081 (6)
C2	0.0351 (7)	0.0496 (9)	0.0490 (8)	−0.0031 (7)	0.0048 (6)	−0.0080 (7)
C3	0.0422 (9)	0.0505 (9)	0.0593 (10)	0.0019 (7)	0.0070 (7)	−0.0073 (8)
C4	0.0533 (10)	0.0649 (12)	0.0645 (12)	−0.0011 (9)	0.0119 (9)	−0.0174 (9)
C5	0.0349 (7)	0.0487 (8)	0.0351 (7)	−0.0025 (6)	0.0041 (6)	−0.0052 (6)
C6	0.0652 (11)	0.0596 (11)	0.0442 (9)	−0.0058 (9)	−0.0023 (8)	−0.0139 (8)
C7	0.0781 (14)	0.0949 (18)	0.0380 (9)	0.0000 (12)	−0.0105 (9)	−0.0129 (10)
C8	0.0703 (13)	0.0866 (16)	0.0436 (9)	0.0039 (12)	−0.0036 (9)	0.0120 (10)
C9	0.0686 (12)	0.0598 (11)	0.0593 (11)	−0.0067 (10)	−0.0079 (9)	0.0123 (9)
C10	0.0547 (10)	0.0495 (9)	0.0448 (8)	−0.0080 (8)	−0.0079 (7)	−0.0001 (7)
C11	0.0340 (7)	0.0454 (8)	0.0352 (7)	−0.0034 (6)	0.0002 (6)	−0.0011 (6)
C12	0.0478 (9)	0.0481 (9)	0.0550 (9)	−0.0091 (7)	0.0060 (7)	−0.0017 (8)
C13	0.0475 (10)	0.0675 (12)	0.0669 (12)	−0.0187 (9)	0.0048 (8)	0.0095 (10)
C14	0.0382 (8)	0.0880 (14)	0.0474 (9)	−0.0055 (9)	0.0064 (7)	0.0101 (9)
C15	0.0404 (8)	0.0797 (13)	0.0446 (9)	0.0077 (9)	0.0035 (7)	−0.0082 (9)
C16	0.0371 (8)	0.0550 (10)	0.0468 (8)	−0.0015 (7)	0.0011 (6)	−0.0085 (7)
C17	0.0308 (7)	0.0353 (7)	0.0404 (7)	0.0023 (5)	−0.0011 (5)	−0.0014 (6)
C18	0.0525 (9)	0.0360 (8)	0.0535 (9)	−0.0028 (7)	−0.0060 (7)	0.0026 (7)
C19	0.0583 (10)	0.0487 (10)	0.0579 (10)	−0.0038 (8)	−0.0057 (8)	0.0175 (8)
C20	0.0462 (9)	0.0618 (11)	0.0423 (8)	0.0039 (8)	−0.0026 (7)	0.0094 (8)
C21	0.0359 (7)	0.0477 (9)	0.0408 (7)	0.0057 (6)	−0.0025 (6)	−0.0054 (6)
C22	0.0312 (7)	0.0360 (7)	0.0409 (7)	0.0042 (6)	0.0012 (5)	−0.0026 (6)
C23	0.0595 (10)	0.0372 (8)	0.0479 (8)	−0.0074 (7)	0.0002 (7)	−0.0047 (7)

Geometric parameters (Å, °)

Cl1—C4	1.802 (2)	C10—H10	0.9300
Cl2—C21	1.7491 (17)	C11—C16	1.384 (2)
O1—C3	1.435 (2)	C11—C12	1.391 (2)
O1—C1	1.4558 (18)	C12—C13	1.392 (3)
N1—C17	1.4414 (18)	C12—H12	0.9300
N1—C1	1.4639 (18)	C13—C14	1.373 (3)
N1—C2	1.4768 (19)	C13—H13	0.9300
C1—C11	1.518 (2)	C14—C15	1.374 (3)
C1—C5	1.539 (2)	C14—H14	0.9300
C2—C3	1.546 (2)	C15—C16	1.387 (2)
C2—H2A	0.9700	C15—H15	0.9300
C2—H2B	0.9700	C16—H16	0.9300
C3—C4	1.503 (3)	C17—C18	1.391 (2)
C3—H3	0.9800	C17—C22	1.407 (2)
C4—H4A	0.9700	C18—C19	1.389 (3)
C4—H4B	0.9700	C18—H18	0.9300
C5—C10	1.386 (2)	C19—C20	1.374 (3)
C5—C6	1.390 (2)	C19—H19	0.9300
C6—C7	1.387 (3)	C20—C21	1.382 (3)
C6—H6	0.9300	C20—H20	0.9300
C7—C8	1.373 (4)	C21—C22	1.402 (2)
C7—H7	0.9300	C22—C23	1.501 (2)
C8—C9	1.378 (3)	C23—H23A	0.9600
C8—H8	0.9300	C23—H23B	0.9600
C9—C10	1.384 (3)	C23—H23C	0.9600
C9—H9	0.9300		
C3—O1—C1	108.45 (12)	C9—C10—H10	119.6
C17—N1—C1	119.47 (12)	C5—C10—H10	119.6
C17—N1—C2	113.23 (12)	C16—C11—C12	119.11 (15)
C1—N1—C2	102.38 (11)	C16—C11—C1	119.70 (14)
O1—C1—N1	105.77 (12)	C12—C11—C1	121.02 (14)
O1—C1—C11	109.11 (12)	C11—C12—C13	119.69 (18)
N1—C1—C11	115.17 (12)	C11—C12—H12	120.2
O1—C1—C5	109.81 (12)	C13—C12—H12	120.2
N1—C1—C5	109.61 (12)	C14—C13—C12	120.57 (18)
C11—C1—C5	107.32 (12)	C14—C13—H13	119.7
N1—C2—C3	103.81 (12)	C12—C13—H13	119.7
N1—C2—H2A	111.0	C13—C14—C15	119.96 (17)
C3—C2—H2A	111.0	C13—C14—H14	120.0
N1—C2—H2B	111.0	C15—C14—H14	120.0
C3—C2—H2B	111.0	C14—C15—C16	120.00 (18)
H2A—C2—H2B	109.0	C14—C15—H15	120.0
O1—C3—C4	107.96 (15)	C16—C15—H15	120.0
O1—C3—C2	104.71 (13)	C11—C16—C15	120.67 (17)
C4—C3—C2	114.58 (16)	C11—C16—H16	119.7
O1—C3—H3	109.8	C15—C16—H16	119.7

C4—C3—H3	109.8	C18—C17—C22	120.61 (14)
C2—C3—H3	109.8	C18—C17—N1	123.27 (14)
C3—C4—Cl1	109.59 (14)	C22—C17—N1	116.11 (13)
C3—C4—H4A	109.8	C19—C18—C17	120.32 (16)
Cl1—C4—H4A	109.8	C19—C18—H18	119.8
C3—C4—H4B	109.8	C17—C18—H18	119.8
Cl1—C4—H4B	109.8	C20—C19—C18	120.32 (16)
H4A—C4—H4B	108.2	C20—C19—H19	119.8
C10—C5—C6	118.17 (16)	C18—C19—H19	119.8
C10—C5—C1	121.31 (14)	C19—C20—C21	119.19 (15)
C6—C5—C1	120.22 (16)	C19—C20—H20	120.4
C7—C6—C5	120.6 (2)	C21—C20—H20	120.4
C7—C6—H6	119.7	C20—C21—C22	122.67 (15)
C5—C6—H6	119.7	C20—C21—Cl2	116.88 (12)
C8—C7—C6	120.58 (18)	C22—C21—Cl2	120.45 (13)
C8—C7—H7	119.7	C21—C22—C17	116.89 (14)
C6—C7—H7	119.7	C21—C22—C23	122.73 (14)
C7—C8—C9	119.31 (19)	C17—C22—C23	120.37 (14)
C7—C8—H8	120.3	C22—C23—H23A	109.5
C9—C8—H8	120.3	C22—C23—H23B	109.5
C8—C9—C10	120.4 (2)	H23A—C23—H23B	109.5
C8—C9—H9	119.8	C22—C23—H23C	109.5
C10—C9—H9	119.8	H23A—C23—H23C	109.5
C9—C10—C5	120.88 (17)	H23B—C23—H23C	109.5

Fig. 1


Cl2
C20
C21
C19
C22
C18
C13
C14
C15
C17
C12
C23
C
N1
C11
C16
C3
O1
C1
C2
C5
C4
Cl1
C6
C10
C9
C7
C8