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Ethyl 2,6-dichloro-4-phenylquinoline-3-carboxylate

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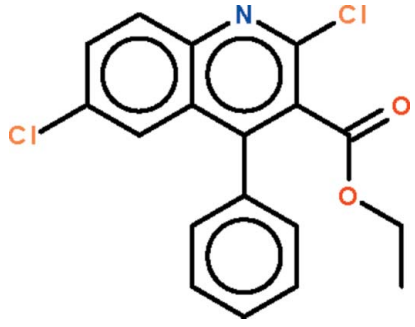
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.144; data-to-parameter ratio = 17.7.

In the title compound, $C_{18}H_{13}Cl_2NO_2$, the quinoline ring system is almost planar (r.m.s. deviation 0.009 Å), and the phenyl and carboxylate planes are twisted away from it by 59.2 (1) and 65.9 (2)°, respectively.

Related literature

The title compound is a 6-chloro analouge of ethyl 2-chloro-4-phenylquinoline-3-carboxylate, which has been examined for endothelin binding affinity; for details, see: Anzini *et al.* (1991, 1992, 2001); Cappelli *et al.* (2008); Pittala *et al.* (2008).



Experimental

Crystal data

$C_{18}H_{13}Cl_2NO_2$
M_r = 346.19
Triclinic, $P\overline{1}$
a = 8.3553 (3) Å
b = 10.1861 (5) Å
c = 10.6731 (6) Å
α = 110.537 (5)°
β = 101.421 (4)°
γ = 95.980 (4)°
V = 818.73 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 295 K
0.34 × 0.26 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.875, T_{max} = 0.906
18102 measured reflections
3700 independent reflections
2537 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.144
S = 1.02
3700 reflections
209 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2954).

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supplementary materials

Acta Cryst. (2009). E**65**, o2982 [**doi:10.1107/S1600536809045334**]

Ethyl 2,6-dichloro-4-phenylquinoline-3-carboxylate

S. M. Roopan, F. N. Khan, M. Vijetha, V. R. Hathwar and S. W. Ng

Experimental

An excess of phosphorus oxychloride (0.9 ml, 10 mmol) and 6-chloro-1,2-dihydro-2-oxo-4-phenylquinoline-3-carboxylate (0.33 g, 1 mmol) were heated for 1 h. The mixture was then added to crushed ice. The solid that formed was collected and recrystallized from methanol.

Refinement

C-bound H-atoms were placed in calculated positions (C-H = 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2–1.5U_{eq}(C). The C—C distance of the ethyl chain was tightly restrained to 1.500 (2) Å.

Figures

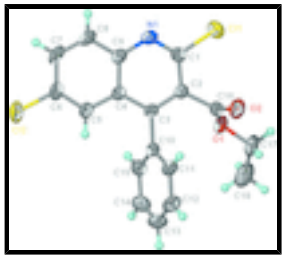


Fig. 1. Displacement ellipsoid plot (Barbour, 2001) of $C_{18}H_{13}Cl_2NO_2$ at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

Ethyl 2,6-dichloro-4-phenylquinoline-3-carboxylate

Crystal data

$C_{18}H_{13}Cl_2NO_2$	$Z = 2$
$M_r = 346.19$	$F_{000} = 356$
Triclinic, $P\overline{1}$	$D_x = 1.404$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.3553$ (3) Å	Cell parameters from 1235 reflections
$b = 10.1861$ (5) Å	$\theta = 1.7$–$21.3°$
$c = 10.6731$ (6) Å	$\mu = 0.40$ mm^{-1}
$\alpha = 110.537$ (5)°	$T = 295$ K
$\beta = 101.421$ (4)°	Block, colourless
$\gamma = 95.980$ (4)°	0.34 × 0.26 × 0.25 mm
$V = 818.73$ (7) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	3700 independent reflections
Radiation source: fine-focus sealed tube	2537 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.031$
$T = 290$ K	$\theta_{max} = 27.5^{o}$
φ and ω scans	$\theta_{min} = 3.4^{o}$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{min} = 0.875$, $T_{max} = 0.906$	$k = -13 \rightarrow 12$
18102 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.144$	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.2927P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
3700 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
209 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.38082 (9)	0.27696 (7)	0.20007 (7)	0.0656 (2)
Cl2	0.09541 (10)	0.80809 (9)	0.87358 (6)	0.0737 (3)
N1	0.2968 (2)	0.38235 (19)	0.42803 (19)	0.0464 (4)
O1	0.4425 (2)	0.66274 (18)	0.20689 (15)	0.0522 (4)
O2	0.2357 (2)	0.48698 (19)	0.05569 (16)	0.0651 (5)
C1	0.3141 (3)	0.4086 (2)	0.3210 (2)	0.0432 (5)
C2	0.2874 (3)	0.5342 (2)	0.2978 (2)	0.0394 (5)
C3	0.2357 (3)	0.6384 (2)	0.3963 (2)	0.0372 (5)
C4	0.2145 (3)	0.6144 (2)	0.5166 (2)	0.0381 (5)
C5	0.1637 (3)	0.7137 (2)	0.6250 (2)	0.0436 (5)
H5	0.1397	0.7989	0.6195	0.052*
C6	0.1503 (3)	0.6832 (3)	0.7373 (2)	0.0479 (5)
C7	0.1808 (3)	0.5550 (3)	0.7485 (2)	0.0534 (6)
H7	0.1695	0.5368	0.8261	0.064*
C8	0.2271 (3)	0.4575 (3)	0.6448 (2)	0.0509 (6)
H8	0.2459	0.3714	0.6512	0.061*

C9	0.2472 (3)	0.4845 (2)	0.5278 (2)	0.0421 (5)
C10	0.2017 (3)	0.7710 (2)	0.3758 (2)	0.0399 (5)
C11	0.0839 (3)	0.7647 (3)	0.2608 (2)	0.0479 (5)
H11	0.0235	0.6769	0.1963	0.057*
C12	0.0565 (3)	0.8896 (3)	0.2421 (3)	0.0607 (7)
H12	−0.0235	0.8855	0.1659	0.073*
C13	0.1478 (4)	1.0192 (3)	0.3363 (3)	0.0650 (7)
H13	0.1301	1.1024	0.3227	0.078*
C14	0.2633 (4)	1.0268 (3)	0.4490 (3)	0.0641 (7)
H14	0.3240	1.1151	0.5123	0.077*
C15	0.2910 (3)	0.9030 (2)	0.4697 (2)	0.0506 (6)
H15	0.3701	0.9088	0.5472	0.061*
C16	0.3163 (3)	0.5558 (2)	0.1716 (2)	0.0443 (5)
C17	0.4740 (4)	0.7060 (3)	0.0971 (3)	0.0749 (9)
H17A	0.3713	0.6852	0.0267	0.090*
H17B	0.5522	0.6533	0.0547	0.090*
C18	0.5432 (6)	0.8615 (3)	0.1550 (4)	0.1137 (15)
H18A	0.5572	0.8916	0.0815	0.170*
H18B	0.6490	0.8806	0.2195	0.170*
H18C	0.4683	0.9129	0.2016	0.170*

Atomic displacement parameters (Å^2)

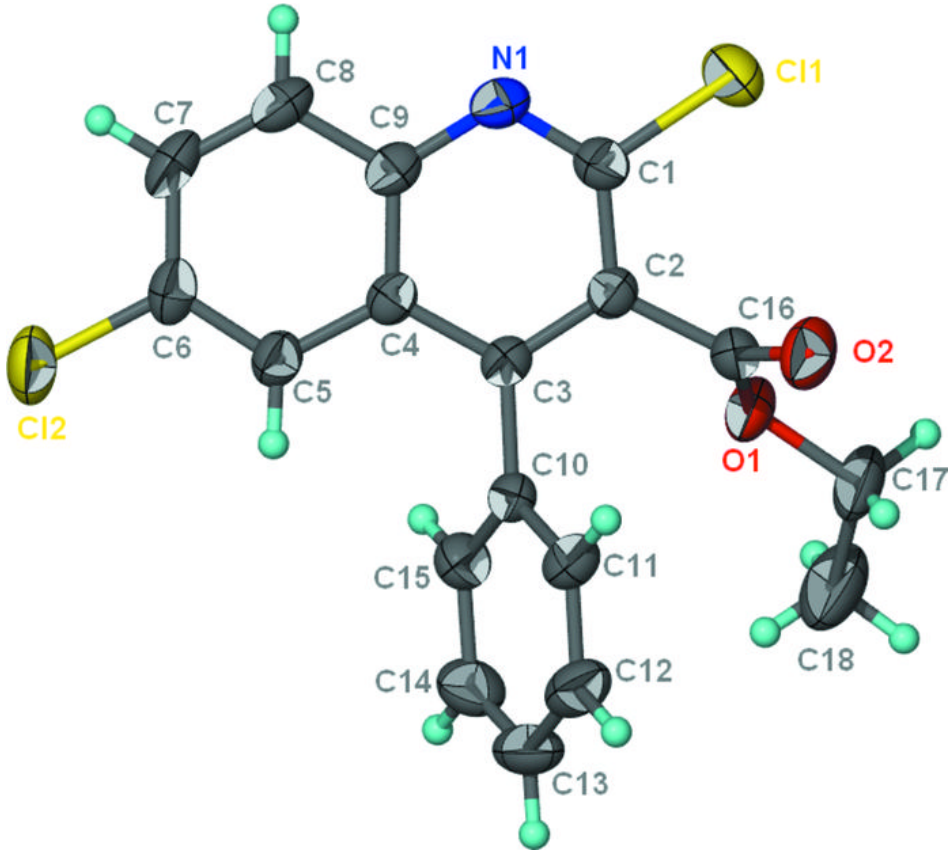
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0792 (5)	0.0546 (4)	0.0702 (4)	0.0224 (3)	0.0354 (4)	0.0209 (3)
Cl2	0.0878 (5)	0.1001 (6)	0.0420 (3)	0.0331 (4)	0.0288 (3)	0.0264 (3)
N1	0.0512 (11)	0.0404 (10)	0.0507 (11)	0.0064 (8)	0.0109 (9)	0.0226 (8)
O1	0.0616 (10)	0.0593 (10)	0.0393 (8)	0.0039 (8)	0.0178 (7)	0.0223 (7)
O2	0.0851 (13)	0.0648 (11)	0.0359 (9)	0.0015 (10)	0.0110 (9)	0.0135 (8)
C1	0.0424 (12)	0.0393 (11)	0.0472 (12)	0.0081 (9)	0.0125 (9)	0.0148 (9)
C2	0.0419 (12)	0.0405 (11)	0.0377 (10)	0.0071 (9)	0.0108 (9)	0.0170 (8)
C3	0.0372 (11)	0.0390 (11)	0.0361 (10)	0.0051 (9)	0.0072 (8)	0.0167 (8)
C4	0.0366 (11)	0.0431 (11)	0.0348 (10)	0.0053 (9)	0.0062 (8)	0.0171 (8)
C5	0.0448 (12)	0.0528 (13)	0.0375 (11)	0.0119 (10)	0.0111 (9)	0.0210 (9)
C6	0.0428 (12)	0.0664 (15)	0.0338 (11)	0.0074 (11)	0.0098 (9)	0.0189 (10)
C7	0.0545 (14)	0.0694 (16)	0.0382 (12)	−0.0051 (12)	0.0054 (10)	0.0309 (11)
C8	0.0577 (15)	0.0511 (13)	0.0479 (13)	0.0015 (11)	0.0063 (11)	0.0298 (11)
C9	0.0406 (12)	0.0441 (11)	0.0423 (11)	0.0017 (9)	0.0065 (9)	0.0212 (9)
C10	0.0461 (12)	0.0414 (11)	0.0398 (11)	0.0128 (9)	0.0176 (9)	0.0193 (9)
C11	0.0505 (14)	0.0519 (13)	0.0484 (12)	0.0093 (10)	0.0132 (10)	0.0272 (10)
C12	0.0577 (16)	0.0768 (19)	0.0698 (16)	0.0248 (14)	0.0205 (13)	0.0486 (15)
C13	0.0747 (19)	0.0517 (15)	0.091 (2)	0.0255 (14)	0.0355 (16)	0.0417 (15)
C14	0.0769 (19)	0.0414 (13)	0.0749 (18)	0.0124 (13)	0.0264 (15)	0.0191 (12)
C15	0.0595 (15)	0.0447 (12)	0.0461 (12)	0.0109 (11)	0.0142 (11)	0.0148 (10)
C16	0.0535 (14)	0.0451 (12)	0.0397 (12)	0.0166 (11)	0.0175 (10)	0.0173 (9)
C17	0.115 (2)	0.0674 (17)	0.0481 (14)	−0.0004 (16)	0.0374 (15)	0.0240 (13)
C18	0.189 (4)	0.082 (2)	0.075 (2)	−0.008 (3)	0.052 (3)	0.0349 (18)

Geometric parameters (Å, °)

Cl1—C1	1.732 (2)	C8—C9	1.407 (3)
Cl2—C6	1.742 (2)	C8—H8	0.93
N1—C1	1.292 (3)	C10—C15	1.385 (3)
N1—C9	1.369 (3)	C10—C11	1.390 (3)
O1—C16	1.325 (3)	C11—C12	1.389 (3)
O1—C17	1.448 (3)	C11—H11	0.93
O2—C16	1.199 (3)	C12—C13	1.375 (4)
C1—C2	1.417 (3)	C12—H12	0.93
C2—C3	1.382 (3)	C13—C14	1.358 (4)
C2—C16	1.499 (3)	C13—H13	0.93
C3—C4	1.429 (3)	C14—C15	1.389 (4)
C3—C10	1.488 (3)	C14—H14	0.93
C4—C5	1.416 (3)	C15—H15	0.93
C4—C9	1.419 (3)	C17—C18	1.485 (2)
C5—C6	1.361 (3)	C17—H17A	0.97
C5—H5	0.93	C17—H17B	0.97
C6—C7	1.397 (4)	C18—H18A	0.96
C7—C8	1.356 (4)	C18—H18B	0.96
C7—H7	0.93	C18—H18C	0.96
C1—N1—C9	117.23 (18)	C11—C10—C3	120.90 (19)
C16—O1—C17	116.36 (18)	C12—C11—C10	120.0 (2)
N1—C1—C2	125.8 (2)	C12—C11—H11	120.0
N1—C1—Cl1	115.05 (16)	C10—C11—H11	120.0
C2—C1—Cl1	119.09 (17)	C13—C12—C11	120.0 (2)
C3—C2—C1	118.10 (19)	C13—C12—H12	120.0
C3—C2—C16	120.40 (18)	C11—C12—H12	120.0
C1—C2—C16	121.50 (19)	C14—C13—C12	120.6 (2)
C2—C3—C4	118.21 (18)	C14—C13—H13	119.7
C2—C3—C10	120.49 (18)	C12—C13—H13	119.7
C4—C3—C10	121.30 (18)	C13—C14—C15	120.1 (3)
C5—C4—C9	118.57 (19)	C13—C14—H14	120.0
C5—C4—C3	123.39 (19)	C15—C14—H14	120.0
C9—C4—C3	118.04 (19)	C10—C15—C14	120.5 (2)
C6—C5—C4	119.4 (2)	C10—C15—H15	119.8
C6—C5—H5	120.3	C14—C15—H15	119.8
C4—C5—H5	120.3	O2—C16—O1	125.1 (2)
C5—C6—C7	122.4 (2)	O2—C16—C2	124.8 (2)
C5—C6—Cl2	119.40 (19)	O1—C16—C2	110.14 (18)
C7—C6—Cl2	118.20 (17)	O1—C17—C18	109.4 (2)
C8—C7—C6	119.2 (2)	O1—C17—H17A	109.8
C8—C7—H7	120.4	C18—C17—H17A	109.8
C6—C7—H7	120.4	O1—C17—H17B	109.8
C7—C8—C9	121.1 (2)	C18—C17—H17B	109.8
C7—C8—H8	119.5	H17A—C17—H17B	108.2
C9—C8—H8	119.5	C17—C18—H18A	109.5
N1—C9—C8	118.0 (2)	C17—C18—H18B	109.5

N1—C9—C4	122.57 (19)	H18A—C18—H18B	109.5
C8—C9—C4	119.4 (2)	C17—C18—H18C	109.5
C15—C10—C11	118.9 (2)	H18A—C18—H18C	109.5
C15—C10—C3	120.20 (19)	H18B—C18—H18C	109.5
C9—N1—C1—C2	0.5 (3)	C7—C8—C9—C4	−1.6 (3)
C9—N1—C1—Cl1	178.84 (15)	C5—C4—C9—N1	179.98 (19)
N1—C1—C2—C3	−1.1 (3)	C3—C4—C9—N1	−0.4 (3)
Cl1—C1—C2—C3	−179.37 (16)	C5—C4—C9—C8	0.5 (3)
N1—C1—C2—C16	178.0 (2)	C3—C4—C9—C8	−179.88 (19)
Cl1—C1—C2—C16	−0.3 (3)	C2—C3—C10—C15	−119.8 (2)
C1—C2—C3—C4	0.8 (3)	C4—C3—C10—C15	60.9 (3)
C16—C2—C3—C4	−178.25 (19)	C2—C3—C10—C11	58.2 (3)
C1—C2—C3—C10	−178.5 (2)	C4—C3—C10—C11	−121.1 (2)
C16—C2—C3—C10	2.4 (3)	C15—C10—C11—C12	−0.5 (3)
C2—C3—C4—C5	179.40 (19)	C3—C10—C11—C12	−178.6 (2)
C10—C3—C4—C5	−1.3 (3)	C10—C11—C12—C13	1.0 (4)
C2—C3—C4—C9	−0.2 (3)	C11—C12—C13—C14	−0.9 (4)
C10—C3—C4—C9	179.14 (19)	C12—C13—C14—C15	0.3 (4)
C9—C4—C5—C6	1.1 (3)	C11—C10—C15—C14	−0.1 (4)
C3—C4—C5—C6	−178.5 (2)	C3—C10—C15—C14	178.0 (2)
C4—C5—C6—C7	−1.7 (3)	C13—C14—C15—C10	0.2 (4)
C4—C5—C6—Cl2	177.61 (16)	C17—O1—C16—O2	5.6 (3)
C5—C6—C7—C8	0.7 (4)	C17—O1—C16—C2	−173.7 (2)
Cl2—C6—C7—C8	−178.63 (18)	C3—C2—C16—O2	−114.0 (3)
C6—C7—C8—C9	1.0 (4)	C1—C2—C16—O2	66.9 (3)
C1—N1—C9—C8	179.8 (2)	C3—C2—C16—O1	65.3 (3)
C1—N1—C9—C4	0.3 (3)	C1—C2—C16—O1	−113.8 (2)
C7—C8—C9—N1	179.0 (2)	C16—O1—C17—C18	148.7 (3)

Fig. 1


N1
Cl1
C8
C7
C9
C1
C2
C16
O2
C6
C4
Cl2
C5
C3
O1
C17
C10
C15
C11
C18
C14
C12
C13