

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

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

S. Mohana Roopan,^a F. Nawaz Khan,^a M. Vijetha,^a Venkatesha R. Hathwar^b and Seik Weng Ng^{c*}

^aChemistry Division, School of Science and Humanities, VIT University, Vellore 632 014, Tamil Nadu, India, ^bSolid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, Karnataka, India, and ^cDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

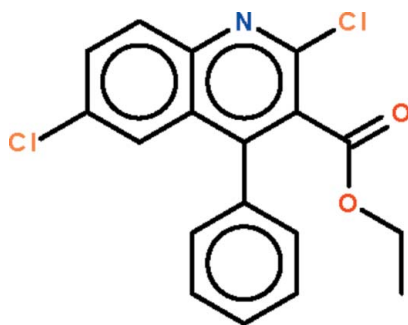
Received 27 October 2009; accepted 29 October 2009

Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.046; wR factor = 0.144; data-to-parameter ratio = 17.7.

In the title compound, $\text{C}_{18}\text{H}_{13}\text{Cl}_2\text{NO}_2$, 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 analogue 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

$\text{C}_{18}\text{H}_{13}\text{Cl}_2\text{NO}_2$
 $M_r = 346.19$

Triclinic, $P\bar{1}$
 $a = 8.3553$ (3) Å

$b = 10.1861$ (5) Å
 $c = 10.6731$ (6) Å
 $\alpha = 110.537$ (5)°
 $\beta = 101.421$ (4)°
 $\gamma = 95.980$ (4)°
 $V = 818.73$ (7) Å³

$Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.40$ mm⁻¹
 $T = 295$ K
 $0.34 \times 0.26 \times 0.25$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

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
 $\Delta\rho_{\text{max}} = 0.41$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.36$ 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).

The authors thank the Department of Science and Technology, India, for use of the diffraction facility set up under the IRHPA–DST programme at IISc. FNK thanks the DST for Fast Track Proposal funding. The authors also thank VIT University and the University of Malaya for supporting this study.

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

References

- Anzini, M., Cappelli, A., Vomero, S., Cagnotto, A. & Skorupska, M. (1992). *Il Farm.* **47**, 191–202.
Anzini, M., Cappelli, A., Vomero, S., Campiani, G., Cagnotto, A. & Skorupska, M. (1991). *Il Farm.* **46**, 1435–1447.
Anzini, M., Cappelli, A., Vomero, S., Seeber, M., Menziani, M. C., Langer, T., Hagen, B., Manzoni, C. & Bourguignon, J.-J. (2001). *J. Med. Chem.* **44**, 1134–1150.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2004). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
Cappelli, A., Giuliani, G., Anzini, M., Riitano, D., Giorgi, G. & Vomero, S. (2008). *Bioorg. Med. Chem.* **16**, 6850–6859.
Pittala, V., Modica, M., Salerno, L., Siracusa, M. A., Guerrero, F., Mereghetti, I., Cagnotto, A., Mennini, T. & Romeo, G. (2008). *Med. Chem.* **4**, 129–137.
Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Westrip, S. P. (2009). publCIF. In preparation.

supplementary materials

Acta Cryst. (2009). E65, 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 1.2–1.5 $U_{eq}(C)$. The C—C distance of the ethyl chain was tightly restrained to 1.500 (2) Å.

Figures

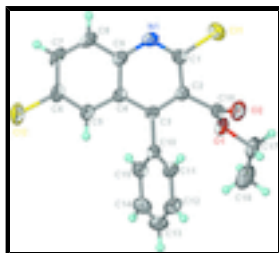


Fig. 1. Displacement ellipsoid plot (Barbour, 2001) of $C_{18}H_{13}Cl_2NO_2$ at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_{18}H_{13}Cl_2NO_2$

$M_r = 346.19$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 8.3553$ (3) Å

$b = 10.1861$ (5) Å

$c = 10.6731$ (6) Å

$\alpha = 110.537$ (5)°

$\beta = 101.421$ (4)°

$\gamma = 95.980$ (4)°

$V = 818.73$ (7) Å³

$Z = 2$

$F_{000} = 356$

$D_x = 1.404$ Mg m⁻³

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

Cell parameters from 1235 reflections

$\theta = 1.7$ – 21.3 °

$\mu = 0.40$ mm⁻¹

$T = 295$ K

Block, colourless

$0.34 \times 0.26 \times 0.25$ mm

supplementary materials

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	$R_{\text{int}} = 0.031$
$T = 290$ K	$\theta_{\text{max}} = 27.5^\circ$
φ and ω scans	$\theta_{\text{min}} = 3.4^\circ$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.875$, $T_{\text{max}} = 0.906$	$k = -13 \rightarrow 12$
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
$wR(F^2) = 0.144$	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.2927P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
3700 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
209 parameters	$\Delta\rho_{\text{max}} = 0.41 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\text{min}} = -0.35 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.38082 (9)	0.27696 (7)	0.20007 (7)	0.0656 (2)
C12	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 (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0792 (5)	0.0546 (4)	0.0702 (4)	0.0224 (3)	0.0354 (4)	0.0209 (3)
C12	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)

supplementary materials

Geometric parameters (\AA , $^\circ$)

C11—C1	1.732 (2)	C8—C9	1.407 (3)
C12—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—C11	115.05 (16)	C10—C11—H11	120.0
C2—C1—C11	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—C12	119.40 (19)	O1—C16—C2	110.14 (18)
C7—C6—C12	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—C11	178.84 (15)	C5—C4—C9—N1	179.98 (19)
N1—C1—C2—C3	-1.1 (3)	C3—C4—C9—N1	-0.4 (3)
C11—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)
C11—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—C12	177.61 (16)	C17—O1—C16—O2	5.6 (3)
C5—C6—C7—C8	0.7 (4)	C17—O1—C16—C2	-173.7 (2)
C12—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)

