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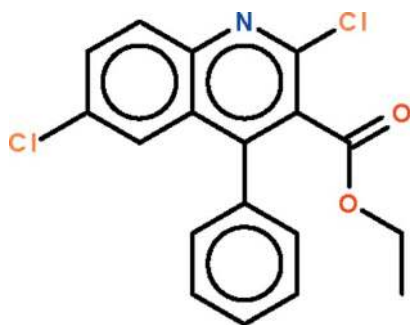
Ethyl 2,6-dichloro-4-phenylquinoline-3-carboxylate

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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ 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 parameters1 restraint
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.41$ e Å⁻³
 $\Delta\rho_{\min} = -0.36$ e Å⁻³Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINTE* (Bruker, 2004); data reduction: *SAINTE*; 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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Ethyl 2,6-dichloro-4-phenylquinoline-3-carboxylate

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S1. 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.

S2. Refinement

C-bound H-atoms were placed in calculated positions ($C-H = 0.93-0.97 \text{ \AA}$) 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 $1.500(2) \text{ \AA}$.

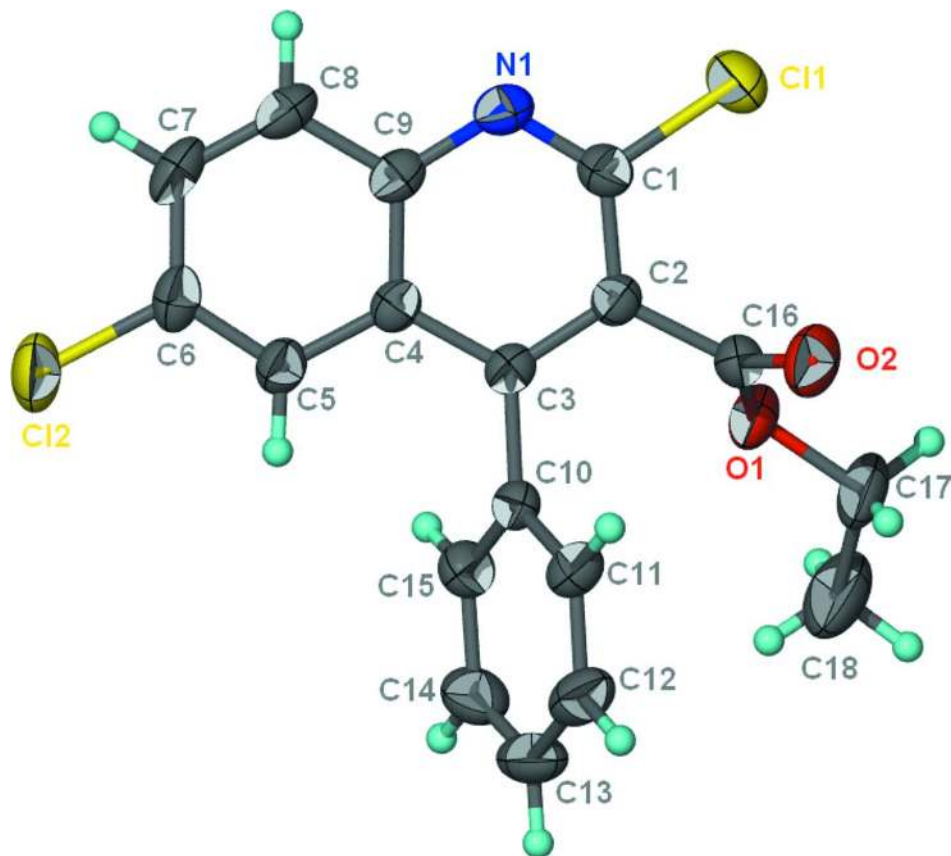


Figure 1

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

C₁₈H₁₃Cl₂NO₂ $M_r = 346.19$ Triclinic, $P\bar{1}$

Hall symbol: -P 1

 $a = 8.3553 (3) \text{ \AA}$ $b = 10.1861 (5) \text{ \AA}$ $c = 10.6731 (6) \text{ \AA}$ $\alpha = 110.537 (5)^\circ$ $\beta = 101.421 (4)^\circ$ $\gamma = 95.980 (4)^\circ$ $V = 818.73 (7) \text{ \AA}^3$ $Z = 2$ $F(000) = 356$ $D_x = 1.404 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1235 reflections

 $\theta = 1.7\text{--}21.3^\circ$ $\mu = 0.40 \text{ mm}^{-1}$ $T = 295 \text{ K}$

Block, colourless

 $0.34 \times 0.26 \times 0.25 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scansAbsorption correction: multi-scan
(SADABS; Sheldrick, 1996) $T_{\min} = 0.875$, $T_{\max} = 0.906$

18102 measured reflections

3700 independent reflections

2537 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.031$ $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.4^\circ$ $h = -10 \rightarrow 10$ $k = -13 \rightarrow 12$ $l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.046$ $wR(F^2) = 0.144$ $S = 1.02$

3700 reflections

209 parameters

1 restraint

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.2927P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.41 \text{ e \AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.36 \text{ e \AA}^{-3}$ Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (Å²)

	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)

Geometric parameters (Å, °)

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)
