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7-Chloro-4-phenethyl-2*H*-1,4-benzoxazin-3(4*H*)-one

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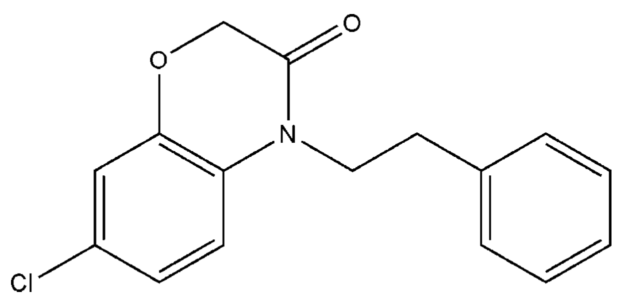
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.049; wR factor = 0.117; data-to-parameter ratio = 11.9.

In the crystal structure of title compound, $C_{16}H_{14}ClNO_2$, the dihedral angle between the aromatic rings is 4.2 (2)°.

Related literature

For related structures, see: Li *et al.* (2008); Zuo *et al.* (2008).



Experimental

Crystal data

$C_{16}H_{14}ClNO_2$
M_r = 287.73
Orthorhombic, *Iba*2
a = 13.528 (4) Å
b = 29.616 (10) Å
c = 7.074 (2) Å
V = 2834.2 (15) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 298 K
0.12 × 0.10 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.968, T_{max} = 0.984
7036 measured reflections
2171 independent reflections
1343 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.117
S = 1.00
2171 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 797 Friedel pairs
Flack parameter: 0.04 (13)

Data collection: *SMART* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); 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: NC2136).

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Zuo, H., Meng, L., Ghate, M., Hwang, K. H., Cho, Y. K., Chandrasekhar, S., Reddy, C. R. & Shin, D. S. (2008). *Tetrahedron Lett.* **49**, 3827–3830.

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7-Chloro-4-phenethyl-2*H*-1,4-benzoxazin-3(4*H*)-one

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S1. Comment

As part of our continuing project on the study of the interactions occurring between small molecules and proteins (Li *et al.*, 2008; Zuo *et al.*, 2008), we report here the synthesis and crystal structure of the title compound. In the crystal structure, the two ring systems are nearly coplanar, the diehderal angle between the aromatic rings being 4.2 (2)°.

S2. Experimental

To the solution of 2-(2,4-dichlorophenoxy)-*N*-phenethylacetamide (0.684 g, 2.0 mmol) in DMF (20 ml), caesium carbonate (0.787 g, 2.4 mmol) was added. The mixture was refluxed for 1.5 h. After completion of the reaction (by TLC monitoring), the DMF was removed under vacuum. Water (20 ml) was added into the residue to obtain a turbid solution and it was extracted by ethyl acetate (20 ml *x* 4). The combined organic layers were washed three times with 10 mL of 1 mol/*L* hydrochloric acid and saturated sodium chloride solution (10 ml *x* 3), dried over $MgSO_4$. And then the mixture was filtered and the filtrate obtained was concentrated under reduced pressure to obtain the corresponding crude product. The product was purified by column chromatography on silica gel using ethyl/acetate = 1/5 as eluent (yield 75%). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solutionof the solid dissolved in ethyl acetate/hexane at room temperature for 10 days.

S3. Refinement

All H atoms were palced in calculated positions and refined as riding, with C—H = 0.93–0.97\%A, and with U_{iso}(H)=1.2Ueq(C). The absolute structure was determined on the basis of 797Friedel pairs.

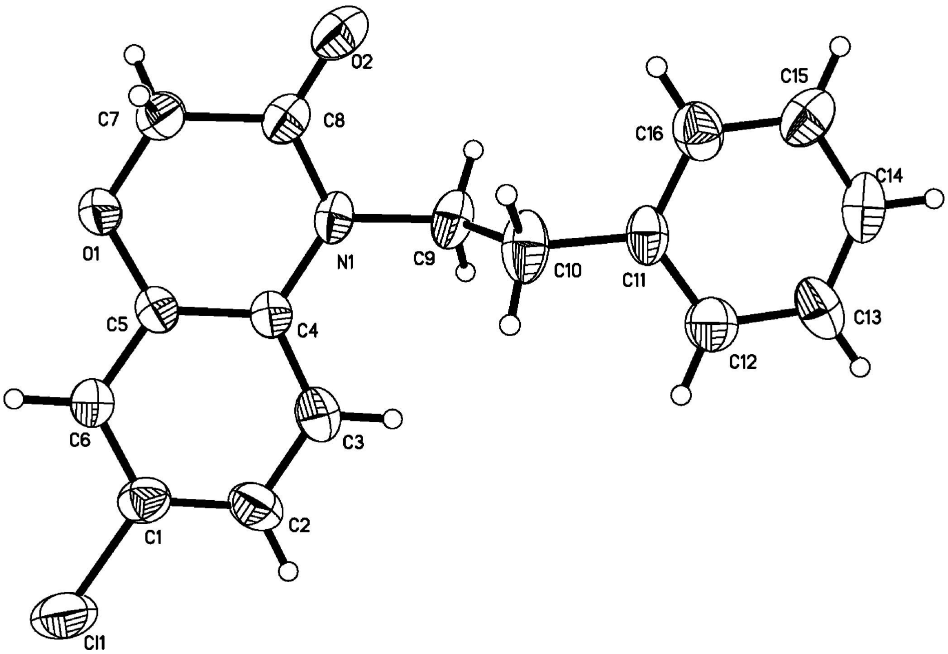

Figure 1

The molecular structure of the title compound with labelling and displacement ellipsoids drawn at the 50% probability level.

7-Chloro-4-phenethyl-2*H*-1,4-benzoxazin-3(4*H*)-one

Crystal data

$C_{16}H_{14}ClNO_2$
M_r = 287.73
Orthorhombic, *Iba*2
Hall symbol: I 2 -2c
a = 13.528 (4) Å
b = 29.616 (10) Å
c = 7.074 (2) Å
V = 2834.2 (15) Å^3
Z = 8
F(000) = 1200
D_x = 1.349 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 809 reflections
θ = 2.6–18.3°
μ = 0.27 mm^{-1}
T = 298 K
Block, colorless
0.12 × 0.10 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and *ω* scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.968, T_{max} = 0.984
7036 measured reflections
2171 independent reflections
1343 reflections with $I > 2\sigma(I)$
R_{int} = 0.067
θ_{max} = 25.0°, θ_{min} = 1.7°
h = −13→16
k = −34→34
l = −8→7

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.117
S = 1.00
2171 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 1.1309P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0057 (5)
Absolute structure: Flack (1983), 797 Friedel pairs
Absolute structure parameter: 0.04 (13)

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 $F^2 > \sigma(F^2)$ 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.The absolute structure was determined on the basis of 800 Friedel pairs.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.42978 (9)	0.23193 (3)	−0.4987 (3)	0.1035 (6)
O1	0.4487 (2)	0.39965 (8)	−0.4216 (4)	0.0762 (10)
O2	0.3501 (2)	0.47560 (9)	−0.0754 (4)	0.0776 (10)
N1	0.3137 (2)	0.40207 (11)	−0.1198 (5)	0.0524 (9)
C1	0.3939 (3)	0.28154 (13)	−0.3869 (7)	0.0641 (12)
C2	0.3273 (4)	0.28028 (16)	−0.2421 (9)	0.0824 (15)
H2	0.3002	0.2530	−0.2033	0.099*
C3	0.3007 (3)	0.32009 (15)	−0.1536 (7)	0.0754 (13)
H3	0.2548	0.3193	−0.0557	0.091*
C4	0.3405 (3)	0.36100 (13)	−0.2074 (6)	0.0489 (10)
C5	0.4080 (3)	0.36097 (13)	−0.3538 (6)	0.0520 (10)
C6	0.4356 (3)	0.32152 (12)	−0.4442 (6)	0.0586 (11)
H6	0.4815	0.3221	−0.5420	0.070*
C7	0.4403 (3)	0.43887 (13)	−0.3107 (7)	0.0608 (11)
H7A	0.4279	0.4640	−0.3956	0.073*
H7B	0.5041	0.4442	−0.2523	0.073*
C8	0.3644 (3)	0.44065 (14)	−0.1590 (6)	0.0556 (11)
C9	0.2368 (3)	0.40386 (14)	0.0274 (6)	0.0640 (11)
H9A	0.1854	0.3822	−0.0027	0.077*
H9B	0.2071	0.4337	0.0278	0.077*
C10	0.2775 (3)	0.39363 (18)	0.2246 (7)	0.0811 (14)
H10A	0.3003	0.3626	0.2285	0.097*
H10B	0.3338	0.4130	0.2491	0.097*
C11	0.2016 (3)	0.40062 (16)	0.3760 (6)	0.0581 (11)
C12	0.1471 (3)	0.36580 (15)	0.4534 (7)	0.0721 (12)
H12	0.1573	0.3364	0.4120	0.087*

C13	0.0770 (3)	0.37441 (18)	0.5926 (8)	0.0776 (14)
H13	0.0415	0.3507	0.6453	0.093*
C14	0.0605 (3)	0.4166 (2)	0.6510 (7)	0.0797 (14)
H14	0.0122	0.4219	0.7419	0.096*
C15	0.1123 (4)	0.45149 (17)	0.5808 (7)	0.0819 (14)
H15	0.1010	0.4807	0.6240	0.098*
C16	0.1827 (3)	0.44326 (15)	0.4432 (7)	0.0724 (12)
H16	0.2185	0.4674	0.3946	0.087*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0963 (9)	0.0644 (7)	0.1497 (14)	−0.0060 (7)	0.0085 (10)	−0.0330 (9)
O1	0.110 (2)	0.0537 (15)	0.064 (2)	−0.0103 (15)	0.038 (2)	−0.0038 (16)
O2	0.088 (2)	0.0731 (19)	0.072 (3)	0.0146 (16)	0.0006 (19)	−0.0206 (18)
N1	0.048 (2)	0.071 (2)	0.038 (2)	0.0065 (17)	0.0033 (17)	−0.0021 (18)
C1	0.058 (2)	0.057 (2)	0.078 (3)	−0.002 (2)	−0.004 (3)	−0.009 (2)
C2	0.079 (3)	0.061 (3)	0.108 (4)	−0.015 (2)	0.015 (3)	0.005 (3)
C3	0.064 (3)	0.079 (3)	0.083 (3)	−0.010 (2)	0.022 (2)	0.005 (3)
C4	0.044 (2)	0.057 (2)	0.046 (2)	0.0034 (19)	0.000 (2)	0.005 (2)
C5	0.050 (2)	0.055 (2)	0.051 (3)	−0.0021 (19)	0.002 (2)	0.007 (2)
C6	0.054 (2)	0.060 (2)	0.062 (3)	0.000 (2)	0.009 (2)	−0.001 (2)
C7	0.064 (2)	0.058 (2)	0.061 (3)	0.006 (2)	0.003 (2)	−0.001 (2)
C8	0.054 (3)	0.064 (3)	0.049 (3)	0.011 (2)	−0.005 (2)	−0.006 (2)
C9	0.051 (2)	0.094 (3)	0.047 (3)	0.016 (2)	0.002 (2)	−0.004 (2)
C10	0.059 (2)	0.132 (4)	0.052 (3)	0.015 (3)	0.002 (2)	0.009 (3)
C11	0.048 (2)	0.087 (3)	0.039 (3)	0.006 (2)	−0.003 (2)	0.007 (2)
C12	0.083 (3)	0.074 (3)	0.059 (3)	0.000 (3)	−0.010 (3)	0.002 (3)
C13	0.071 (3)	0.097 (3)	0.065 (3)	−0.023 (3)	0.000 (3)	0.021 (3)
C14	0.063 (3)	0.127 (4)	0.049 (3)	0.001 (3)	0.006 (2)	0.000 (3)
C15	0.089 (3)	0.088 (3)	0.069 (3)	0.007 (3)	−0.001 (3)	−0.023 (3)
C16	0.070 (3)	0.082 (3)	0.066 (3)	−0.021 (2)	0.003 (3)	0.000 (3)

Geometric parameters (Å, º)

Cl1—C1	1.737 (4)	C7—H7B	0.9700
O1—C5	1.359 (4)	C9—C10	1.529 (6)
O1—C7	1.406 (4)	C9—H9A	0.9700
O2—C8	1.208 (4)	C9—H9B	0.9700
N1—C8	1.361 (5)	C10—C11	1.498 (6)
N1—C4	1.413 (5)	C10—H10A	0.9700
N1—C9	1.473 (5)	C10—H10B	0.9700
C1—C2	1.365 (7)	C11—C16	1.373 (5)
C1—C6	1.372 (5)	C11—C12	1.381 (6)
C2—C3	1.383 (6)	C12—C13	1.390 (6)
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.380 (5)	C13—C14	1.335 (6)
C3—H3	0.9300	C13—H13	0.9300

C4—C5	1.381 (5)	C14—C15	1.344 (6)
C5—C6	1.383 (5)	C14—H14	0.9300
C6—H6	0.9300	C15—C16	1.383 (6)
C7—C8	1.486 (5)	C15—H15	0.9300
C7—H7A	0.9700	C16—H16	0.9300

C5—O1—C7	117.8 (3)	N1—C9—C10	112.6 (3)
C8—N1—C4	120.2 (3)	N1—C9—H9A	109.1
C8—N1—C9	118.0 (3)	C10—C9—H9A	109.1
C4—N1—C9	121.5 (3)	N1—C9—H9B	109.1
C2—C1—C6	121.1 (4)	C10—C9—H9B	109.1
C2—C1—Cl1	120.2 (4)	H9A—C9—H9B	107.8
C6—C1—Cl1	118.7 (4)	C11—C10—C9	112.2 (3)
C1—C2—C3	119.3 (4)	C11—C10—H10A	109.2
C1—C2—H2	120.4	C9—C10—H10A	109.2
C3—C2—H2	120.4	C11—C10—H10B	109.2
C4—C3—C2	121.5 (4)	C9—C10—H10B	109.2
C4—C3—H3	119.3	H10A—C10—H10B	107.9
C2—C3—H3	119.3	C16—C11—C12	116.7 (4)
C3—C4—C5	117.7 (4)	C16—C11—C10	120.1 (4)
C3—C4—N1	122.3 (4)	C12—C11—C10	123.1 (5)
C5—C4—N1	120.0 (3)	C11—C12—C13	120.5 (4)
O1—C5—C4	122.1 (4)	C11—C12—H12	119.7
O1—C5—C6	116.1 (4)	C13—C12—H12	119.7
C4—C5—C6	121.7 (4)	C14—C13—C12	120.4 (4)
C1—C6—C5	118.8 (4)	C14—C13—H13	119.8
C1—C6—H6	120.6	C12—C13—H13	119.8
C5—C6—H6	120.6	C13—C14—C15	121.2 (5)
O1—C7—C8	119.2 (3)	C13—C14—H14	119.4
O1—C7—H7A	107.5	C15—C14—H14	119.4
C8—C7—H7A	107.5	C14—C15—C16	118.9 (5)
O1—C7—H7B	107.5	C14—C15—H15	120.5
C8—C7—H7B	107.5	C16—C15—H15	120.5
H7A—C7—H7B	107.0	C11—C16—C15	122.3 (4)
O2—C8—N1	122.6 (4)	C11—C16—H16	118.9
O2—C8—C7	119.7 (4)	C15—C16—H16	118.9
N1—C8—C7	117.8 (4)		

C6—C1—C2—C3	0.8 (7)	C4—N1—C8—O2	−173.2 (4)
Cl1—C1—C2—C3	179.2 (4)	C9—N1—C8—O2	1.6 (6)
C1—C2—C3—C4	−0.6 (8)	C4—N1—C8—C7	6.6 (5)
C2—C3—C4—C5	0.3 (7)	C9—N1—C8—C7	−178.6 (3)
C2—C3—C4—N1	179.5 (4)	O1—C7—C8—O2	−172.8 (4)
C8—N1—C4—C3	171.0 (4)	O1—C7—C8—N1	7.4 (6)
C9—N1—C4—C3	−3.6 (6)	C8—N1—C9—C10	−89.3 (5)
C8—N1—C4—C5	−9.8 (5)	C4—N1—C9—C10	85.4 (5)
C9—N1—C4—C5	175.6 (3)	N1—C9—C10—C11	173.5 (4)
C7—O1—C5—C4	15.7 (5)	C9—C10—C11—C16	−81.2 (6)

C7—O1—C5—C6	−166.4 (4)	C9—C10—C11—C12	98.2 (5)
C3—C4—C5—O1	177.5 (4)	C16—C11—C12—C13	−0.1 (6)
N1—C4—C5—O1	−1.7 (5)	C10—C11—C12—C13	−179.5 (4)
C3—C4—C5—C6	−0.2 (6)	C11—C12—C13—C14	1.1 (7)
N1—C4—C5—C6	−179.5 (4)	C12—C13—C14—C15	−1.6 (8)
C2—C1—C6—C5	−0.8 (7)	C13—C14—C15—C16	1.1 (8)
Cl1—C1—C6—C5	−179.2 (3)	C12—C11—C16—C15	−0.4 (7)
O1—C5—C6—C1	−177.4 (4)	C10—C11—C16—C15	179.0 (4)
C4—C5—C6—C1	0.5 (6)	C14—C15—C16—C11	−0.1 (7)
C5—O1—C7—C8	−18.3 (5)		