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3-(2,4-Dichlorophenyl)-1-phenyl-2,3-dihydro-1*H*-naphtho[1,2-e][1,3]oxazine

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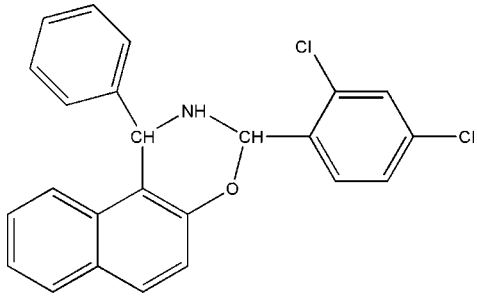
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.104; data-to-parameter ratio = 14.8.

In the title compound, $C_{24}H_{17}Cl_2NO$, the oxazine ring adopts a half-chair conformation. The dihedral angle between the phenyl ring and the naphthyl ring system is 78.56 (9)°. Intramolecular C—H···Cl hydrogen bonding is observed in the crystal structure.

Related literature

For general background, see: Fuganti *et al.* (1994); Ren *et al.* (2001).



Experimental

Crystal data

$C_{24}H_{17}Cl_2NO$
M_r = 406.29
Triclinic, $P\overline{1}$
a = 6.664 (3) Å
b = 8.224 (3) Å
c = 18.106 (7) Å
α = 92.269 (6)°
β = 99.420 (5)°
γ = 98.624 (6)°
V = 965.8 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 291 (2) K
0.30 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
7267 measured reflections
3783 independent reflections
3386 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.104
S = 1.02
3783 reflections
256 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C18—H18···Cl1	0.98	2.60	3.030 (3)	107

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2366).

References

Bruker (2000). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fuganti, C., Lanati, S. & Servi, S. (1994). *Bioorg. Med. Chem.* **4**, 723–726.
Ren, H., Grady, S., Gamenara, D., Heinzen, H., Moyna, P., Croft, S. L., Kendrick, H., Yardley, V. & Moyna, G. (2001). *Bioorg. Med. Chem. Lett.* **11**, 1851–1854.

supplementary materials

Acta Cryst. (2007). E**63**, o4781 [**doi:10.1107/S160053680705787X**]

3-(2,4-Dichlorophenyl)-1-phenyl-2,3-dihydro-1*H*-naphtho[1,2-*e*][1,3]oxazine

Z.-G. Yin, H.-Y. Qian, C. Yu-Zhen and F. Yu-Li

Comment

The class of oxazine derivatives is useful heterocyclic compound which is widely used as antimalarial agent (Ren *et al.*, 2001) and a versatile intermediate for the synthesis of carbapenems (Fuganti *et al.*, 1994). Here we present the synthesis and crystal structure of the title compound.

In the molecule (Fig. 1), the oxazine ring is distorted and adopts a half chair conformation, O1 and N1 atoms deviate from the O1—C18—N1—C11—C1—C2 mean plane by 0.168 (1) and 0.282 (2)%A, respectively. The dihedral angle between the C12-phenyl ring and naphthyl system is 78.56 (9)°. Intra-molecular C—H···Cl hydrogen bond is observed in the crystal structure (Table 1), but no inter-molecular hydrogen bonding occurs in the crystal structure.

Experimental

1-(Amino(phenyl)methyl)naphthalen-2-ol (1 mmol, 0.249 g) was dissolved in anhydrous methanol, the solution was stirred for several min. and then 2,4-dichlorobenzyaldehyde (1 mmol 0.175 g) in methanol (8 ml) was added dropwise and the mixture was stirred at room temperature for 2 h. The product was isolated and recrystallized in a methanol solution, colourless single crystals were obtained after 2 d.

Refinement

Amine H atom was located in a difference Fourier map and positional parameters were refined, U_{iso}(H) = 1.2U_{eq}(N). Other H atoms were placed in calculated positions with C—H = 0.93 Å (aromatic) and 0.97Å (methine) and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C).

Figures

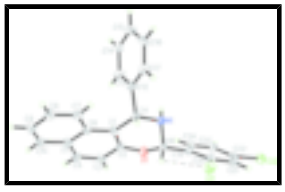


Fig. 1. the *ORTEP* plot of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as small spheres of arbitrary radii.

3-(2,4-Dichlorophenyl)-1-phenyl-2,3-dihydro-1*H*-naphtho[1,2-e][1,3]oxazine

Crystal data

$C_{24}H_{17}Cl_2NO$	Z = 2
M_r = 406.29	F_{000} = 420

Triclinic, $P\bar{1}$	D_x = 1.397 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 6.664 (3) Å	Cell parameters from 816 reflections
b = 8.224 (3) Å	θ = 2.3–20.1º
c = 18.106 (7) Å	μ = 0.35 mm^{-1}
α = 92.269 (6)º	T = 291 (2) K
β = 99.420 (5)º	Block, colourless
γ = 98.624 (6)º	0.30 × 0.26 × 0.24 mm
V = 965.8 (7) Å^3	

Data collection

Bruker SMART Apex CCD area-detector diffractometer	3386 reflections with $I > 2\sigma(I)$
Radiation source: sealed tube	R_{int} = 0.034
Monochromator: graphite	θ_{max} = 26.0º
T = 298(2) K	θ_{min} = 1.1º
φ and ω scans	h = −8→8
Absorption correction: none	k = −10→10
7267 measured reflections	l = −22→22
3783 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.047	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.104	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.66P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max} < 0.001$
3783 reflections	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
256 parameters	$\Delta\rho_{min}$ = −0.20 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 calculat-

ing *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}
C1	−0.0160 (3)	0.4644 (2)	0.26512 (12)	0.0363 (4)
C2	−0.1740 (3)	0.5189 (2)	0.21512 (10)	0.0317 (4)
C3	−0.3762 (3)	0.4255 (3)	0.19891 (12)	0.0378 (5)
H3	−0.4789	0.4654	0.1669	0.045*
C4	−0.4207 (3)	0.2810 (3)	0.22920 (13)	0.0437 (5)
H4	−0.5538	0.2222	0.2192	0.052*
C5	−0.2576 (3)	0.2171 (3)	0.27800 (12)	0.0381 (5)
C6	−0.0633 (3)	0.3062 (2)	0.29281 (11)	0.0350 (4)
C7	0.1011 (4)	0.2367 (3)	0.33856 (12)	0.0440 (5)
H7	0.2338	0.2958	0.3506	0.053*
C8	0.0552 (3)	0.0841 (3)	0.36297 (12)	0.0395 (5)
H8	0.1598	0.0375	0.3906	0.047*
C9	−0.1457 (4)	−0.0061 (3)	0.34780 (14)	0.0481 (6)
H9	−0.1714	−0.1090	0.3669	0.058*
C10	−0.3037 (4)	0.0551 (3)	0.30547 (13)	0.0465 (6)
H10	−0.4359	−0.0053	0.2947	0.056*
C11	0.1908 (3)	0.5714 (3)	0.28337 (11)	0.0341 (4)
H11	0.2941	0.5128	0.2663	0.041*
C12	0.2558 (3)	0.6199 (3)	0.36802 (12)	0.0388 (5)
C13	0.1190 (3)	0.6381 (3)	0.41515 (11)	0.0408 (5)
H13	−0.0217	0.6114	0.3975	0.049*
C14	0.1890 (4)	0.6959 (3)	0.48886 (13)	0.0470 (5)
H14	0.0935	0.7079	0.5200	0.056*
C15	0.3972 (4)	0.7365 (3)	0.51786 (13)	0.0481 (6)
H15	0.4417	0.7756	0.5676	0.058*
C16	0.5396 (4)	0.7171 (3)	0.46977 (14)	0.0554 (7)
H16	0.6801	0.7459	0.4874	0.066*
C17	0.4700 (3)	0.6547 (3)	0.39579 (12)	0.0391 (5)
H17	0.5638	0.6360	0.3648	0.047*
C18	0.0708 (3)	0.7100 (2)	0.17348 (11)	0.0332 (4)
H18	0.1195	0.6285	0.1427	0.040*
C19	0.0854 (3)	0.8725 (2)	0.13725 (11)	0.0356 (4)
C20	0.2413 (3)	0.9202 (3)	0.09702 (12)	0.0409 (5)
C21	0.2598 (4)	1.0652 (3)	0.06420 (12)	0.0419 (5)
H21	0.3635	1.0925	0.0361	0.050*
C22	0.1272 (3)	1.1695 (2)	0.07259 (11)	0.0333 (4)
C23	−0.0264 (3)	1.1347 (3)	0.11365 (13)	0.0419 (5)
H23	−0.1131	1.2106	0.1207	0.050*
C24	−0.0500 (3)	0.9809 (3)	0.14506 (12)	0.0410 (5)
H24	−0.1576	0.9520	0.1713	0.049*
Cl1	0.41832 (9)	0.78745 (8)	0.08509 (4)	0.05364 (18)
Cl2	0.15778 (9)	1.36256 (7)	0.03317 (3)	0.04927 (17)

N1	0.1937 (3)	0.7299 (2)	0.24802 (10)	0.0359 (4)
H1A	0.138 (4)	0.797 (3)	0.2759 (14)	0.043*
O1	−0.1441 (2)	0.65590 (19)	0.17832 (8)	0.0403 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0295 (10)	0.0336 (10)	0.0448 (12)	0.0019 (8)	0.0074 (8)	−0.0025 (9)
C2	0.0261 (9)	0.0415 (11)	0.0264 (9)	−0.0008 (8)	0.0073 (7)	−0.0010 (8)
C3	0.0324 (10)	0.0398 (11)	0.0389 (11)	−0.0012 (9)	0.0066 (8)	−0.0008 (9)
C4	0.0298 (10)	0.0464 (12)	0.0520 (13)	−0.0084 (9)	0.0130 (9)	−0.0001 (10)
C5	0.0294 (10)	0.0428 (11)	0.0399 (11)	−0.0066 (8)	0.0131 (8)	−0.0070 (9)
C6	0.0372 (11)	0.0364 (10)	0.0286 (10)	−0.0029 (8)	0.0072 (8)	−0.0038 (8)
C7	0.0546 (14)	0.0467 (12)	0.0306 (10)	0.0130 (10)	0.0027 (9)	0.0034 (9)
C8	0.0362 (11)	0.0452 (12)	0.0384 (11)	0.0061 (9)	0.0113 (9)	0.0015 (9)
C9	0.0502 (14)	0.0424 (12)	0.0492 (13)	−0.0049 (10)	0.0120 (11)	0.0029 (10)
C10	0.0428 (13)	0.0446 (12)	0.0473 (13)	−0.0130 (10)	0.0128 (10)	−0.0017 (10)
C11	0.0282 (10)	0.0416 (11)	0.0278 (9)	−0.0028 (8)	−0.0031 (7)	0.0114 (8)
C12	0.0349 (11)	0.0468 (12)	0.0351 (11)	0.0109 (9)	0.0012 (8)	0.0087 (9)
C13	0.0397 (11)	0.0541 (13)	0.0303 (10)	0.0138 (10)	0.0047 (9)	0.0020 (9)
C14	0.0559 (14)	0.0395 (12)	0.0411 (12)	−0.0034 (10)	0.0054 (10)	0.0003 (9)
C15	0.0543 (14)	0.0510 (13)	0.0362 (11)	0.0067 (11)	0.0030 (10)	−0.0071 (10)
C16	0.0403 (13)	0.0630 (16)	0.0516 (14)	−0.0134 (11)	0.0005 (11)	−0.0193 (12)
C17	0.0329 (11)	0.0475 (12)	0.0421 (12)	0.0208 (9)	0.0076 (9)	0.0070 (9)
C18	0.0310 (10)	0.0354 (10)	0.0316 (10)	0.0033 (8)	0.0008 (8)	0.0064 (8)
C19	0.0369 (11)	0.0313 (10)	0.0360 (10)	0.0015 (8)	0.0027 (8)	0.0005 (8)
C20	0.0409 (12)	0.0381 (11)	0.0424 (12)	0.0029 (9)	0.0059 (9)	0.0060 (9)
C21	0.0537 (13)	0.0375 (11)	0.0325 (11)	0.0102 (10)	−0.0021 (9)	0.0046 (9)
C22	0.0375 (10)	0.0315 (10)	0.0260 (9)	0.0023 (8)	−0.0057 (8)	−0.0001 (7)
C23	0.0423 (12)	0.0381 (11)	0.0477 (13)	0.0094 (9)	0.0119 (10)	0.0037 (9)
C24	0.0427 (12)	0.0519 (13)	0.0314 (10)	0.0115 (10)	0.0109 (9)	0.0054 (9)
Cl1	0.0490 (3)	0.0618 (4)	0.0551 (4)	0.0125 (3)	0.0175 (3)	0.0144 (3)
Cl2	0.0547 (3)	0.0395 (3)	0.0520 (3)	0.0126 (2)	−0.0025 (3)	0.0124 (2)
N1	0.0295 (9)	0.0434 (10)	0.0324 (9)	−0.0014 (7)	0.0026 (7)	0.0111 (7)
O1	0.0280 (7)	0.0484 (9)	0.0420 (8)	0.0047 (6)	−0.0019 (6)	0.0125 (7)

Geometric parameters (Å, °)

C1—C2	1.411 (3)	C13—H13	0.9300
C1—C6	1.422 (3)	C14—C15	1.386 (3)
C1—C11	1.499 (3)	C14—H14	0.9300
C2—O1	1.337 (2)	C15—C16	1.411 (4)
C2—C3	1.426 (3)	C15—H15	0.9300
C3—C4	1.342 (3)	C16—C17	1.394 (3)
C3—H3	0.9300	C16—H16	0.9300
C4—C5	1.460 (3)	C17—H17	0.9300
C4—H4	0.9300	C18—N1	1.449 (3)
C5—C6	1.368 (3)	C18—O1	1.453 (2)
C5—C10	1.448 (3)	C18—C19	1.509 (3)

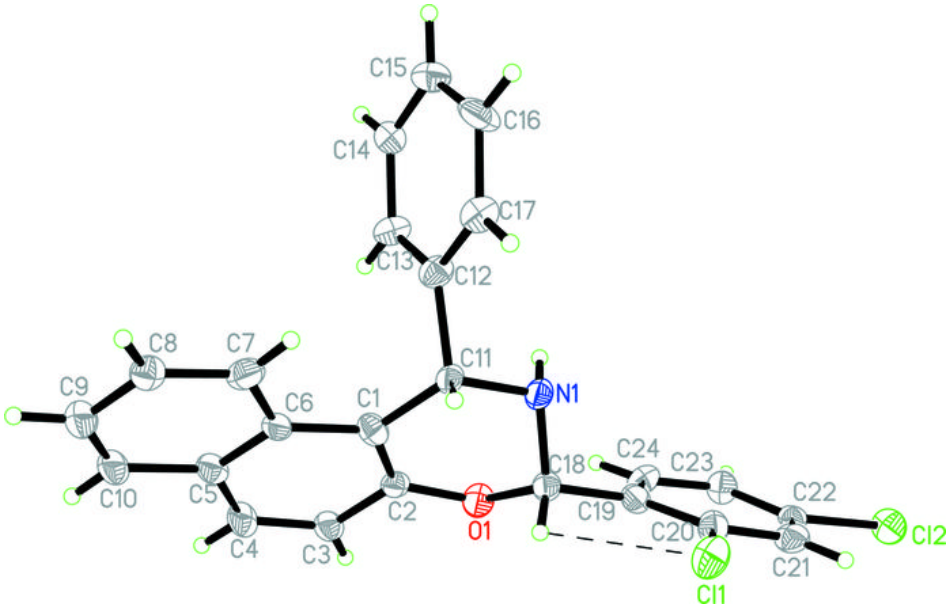
C6—C7	1.463 (3)	C18—H18	0.9800
C7—C8	1.355 (3)	C19—C24	1.379 (3)
C7—H7	0.9300	C19—C20	1.383 (3)
C8—C9	1.409 (3)	C20—C21	1.351 (3)
C8—H8	0.9300	C20—Cl1	1.756 (2)
C9—C10	1.369 (4)	C21—C22	1.342 (3)
C9—H9	0.9300	C21—H21	0.9300
C10—H10	0.9300	C22—C23	1.364 (3)
C11—N1	1.474 (3)	C22—Cl2	1.764 (2)
C11—C12	1.542 (3)	C23—C24	1.405 (3)
C11—H11	0.9800	C23—H23	0.9300
C12—C13	1.366 (3)	C24—H24	0.9300
C12—C17	1.416 (3)	N1—H1A	0.89 (2)
C13—C14	1.380 (3)		
C2—C1—C6	116.83 (18)	C14—C13—H13	119.9
C2—C1—C11	118.91 (18)	C13—C14—C15	122.0 (2)
C6—C1—C11	124.21 (19)	C13—C14—H14	119.0
O1—C2—C1	123.06 (17)	C15—C14—H14	119.0
O1—C2—C3	115.88 (18)	C14—C15—C16	118.2 (2)
C1—C2—C3	121.02 (19)	C14—C15—H15	120.9
C4—C3—C2	121.0 (2)	C16—C15—H15	120.9
C4—C3—H3	119.5	C17—C16—C15	120.1 (2)
C2—C3—H3	119.5	C17—C16—H16	120.0
C3—C4—C5	119.18 (19)	C15—C16—H16	120.0
C3—C4—H4	120.4	C16—C17—C12	119.6 (2)
C5—C4—H4	120.4	C16—C17—H17	120.2
C6—C5—C10	121.7 (2)	C12—C17—H17	120.2
C6—C5—C4	119.6 (2)	N1—C18—O1	109.70 (16)
C10—C5—C4	118.57 (18)	N1—C18—C19	110.19 (16)
C5—C6—C1	122.0 (2)	O1—C18—C19	107.78 (16)
C5—C6—C7	119.0 (2)	N1—C18—H18	109.7
C1—C6—C7	118.94 (19)	O1—C18—H18	109.7
C8—C7—C6	118.3 (2)	C19—C18—H18	109.7
C8—C7—H7	120.8	C24—C19—C20	117.6 (2)
C6—C7—H7	120.8	C24—C19—C18	121.76 (19)
C7—C8—C9	122.1 (2)	C20—C19—C18	120.58 (19)
C7—C8—H8	118.9	C21—C20—C19	122.2 (2)
C9—C8—H8	118.9	C21—C20—Cl1	118.04 (18)
C10—C9—C8	121.2 (2)	C19—C20—Cl1	119.74 (17)
C10—C9—H9	119.4	C22—C21—C20	119.4 (2)
C8—C9—H9	119.4	C22—C21—H21	120.3
C9—C10—C5	117.6 (2)	C20—C21—H21	120.3
C9—C10—H10	121.2	C21—C22—C23	122.2 (2)
C5—C10—H10	121.2	C21—C22—Cl2	119.43 (17)
N1—C11—C1	112.31 (16)	C23—C22—Cl2	118.29 (16)
N1—C11—C12	104.43 (17)	C22—C23—C24	118.1 (2)
C1—C11—C12	112.58 (17)	C22—C23—H23	121.0
N1—C11—H11	109.1	C24—C23—H23	121.0
C1—C11—H11	109.1	C19—C24—C23	120.4 (2)

C12—C11—H11	109.1	C19—C24—H24	119.8
C13—C12—C17	119.7 (2)	C23—C24—H24	119.8
C13—C12—C11	123.50 (19)	C18—N1—C11	111.41 (16)
C17—C12—C11	116.68 (19)	C18—N1—H1A	107.6 (16)
C12—C13—C14	120.2 (2)	C11—N1—H1A	108.0 (16)
C12—C13—H13	119.9	C2—O1—C18	113.20 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C18—H18···Cl1	0.98	2.60	3.030 (3)	107

Fig. 1


C15
C16
C14
C17
C13
C12
C8
C7
C11
C9
C6
C1
N1
C24
C23
C18
C22
C10
C5
C2
O1
C19
Cl2
C20
C4
C3
C21
Cl1