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(±)-3-(2,6-Dichlorophenyl)-1-phenyl-2,3-dihydro-1*H*-naphtho[1,2-e][1,3]-oxazine

Zhi-Gang Yin,* Heng-Yu Qian, Chen Yu-Zhen and Feng Yu-Li

Key Laboratory of Surface and Interface Science of Henan, School of Material and Chemical Engineering, Zhengzhou University of Light Industry, Zhengzhou 450002, People's Republic of China
Correspondence e-mail: yinzhigang3141@yahoo.com.cn

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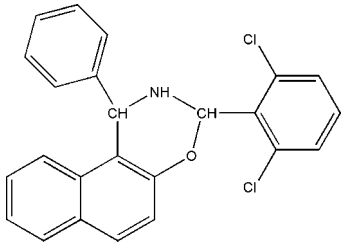
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.050; wR factor = 0.151; data-to-parameter ratio = 15.8.

In the title compound, $C_{24}H_{17}Cl_2NO$, the oxazine ring is distorted and adopts an envelope conformation. The dihedral angle between the dichlorophenyl ring and the naphthyl system is 85.30 (13)°. An intramolecular N—H···Cl hydrogen bond is observed.

Related literature

For related literature, see: Cremer & Pople (1975); Fuganti *et al.* (1994); Ren *et al.* (2001).



Experimental

Crystal data

$C_{24}H_{17}Cl_2NO$
M_r = 406.29
Orthorhombic, *Pbca*
a = 10.8041 (9) Å
b = 10.0189 (9) Å
c = 36.119 (3) Å
V = 3909.7 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 298 (2) K
0.27 × 0.23 × 0.22 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.912, T_{max} = 0.928
43758 measured reflections
4060 independent reflections
2484 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.151
S = 1.05
4060 reflections
257 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl2	0.851 (10)	2.88 (2)	3.406 (2)	121.6 (19)

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: DN2255).

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supplementary materials

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(±)-3-(2,6-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(I).

The oxazine ring is distorted and adopts a half chair conformation(Fig.1), the dihedral angle between the O1/C15/C16 plane and the N1/C1/C7/C8 plane is 46.08°.

The ring puckering parameters (Cremer & Pople, 1975) for the oxazine are, Q = 0.450 (2) Å, θ = 125.0 (3) ° and φ = 298.2 (3) ° indicating an enveloppe conformation. The dichlorophenyl ring makes dihedral angles of 61.27 (18)° and 61.35 (15)° with the benzene ring and the naphthyl system, respectively. The C7—O1 and C7—N1 bond lengths are 1.454 (3)Å and 1.429 (3)\%A. The structure is stabilized by intramolecular N1—H1···Cl2 hydrogen bond(table 1).

Experimental

1-(amino(phenyl)methyl)naphthalen-2-ol(1 mmol, 0.249 g) was dissolved in anhydrous methanol, the mixture was stirred for several minitutes, 2,6-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 methanol, colourless single crystals of (I) was obtained after 3 d.

Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.98 Å (methine) with U_{iso}(H) = 1.2U_{eq}(C). H atoms attached to nitrogen has been located in difference Fourier maps and included in the subsequent refinement using restraints (N—H= 0.86 (1)Å with U_{iso}(H) = 1.5U_{eq}(N).

Figures

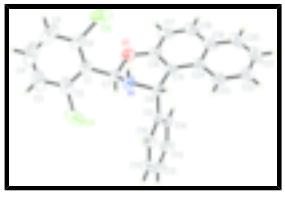


Fig. 1. Molecular view of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as small spheres of arbitrary radii.

(+/-)-3-(2,6-Dichlorophenyl)-1-phenyl-2,3-dihydro-1*H*-naphtho[1,2-\ e][1,3]oxazine

Crystal data

$C_{24}H_{17}Cl_2NO$	F_{000} = 1680
M_r = 406.29	D_x = 1.380 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 1420 reflections
a = 10.8041 (9) Å	θ = 2.2–24.3º
b = 10.0189 (9) Å	μ = 0.35 mm^{-1}
c = 36.119 (3) Å	*T* = 298 (2) K
V = 3909.7 (6) Å^3	Block, colourless
Z = 8	0.27 × 0.23 × 0.22 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	4060 independent reflections
Radiation source: sealed tube	2484 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.047
T = 298(2) K	θ_{max} = 26.5º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	*h* = −13→13
T_{min} = 0.912, T_{max} = 0.928	*k* = −12→12
43758 measured reflections	*l* = −45→45

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.050	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.151	$w = 1/[\sigma^2(F_o^2) + (0.0627P)^2 + 1.1294P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001
4060 reflections	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
257 parameters	$\Delta\rho_{min}$ = −0.40 e Å^{-3}
1 restraint	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0016 (4)

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 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.35756 (10)	−0.01015 (9)	0.47316 (3)	0.1301 (4)
Cl2	−0.05546 (8)	0.25328 (10)	0.42897 (3)	0.1288 (4)
O1	0.09492 (15)	0.05472 (17)	0.39088 (5)	0.0714 (5)
N1	0.24544 (18)	0.23064 (18)	0.40158 (5)	0.0577 (5)
H1	0.1936 (18)	0.2944 (18)	0.4028 (7)	0.069*
C1	0.1477 (2)	0.1169 (2)	0.45289 (6)	0.0616 (6)
C2	0.2172 (3)	0.0667 (3)	0.48184 (8)	0.0848 (9)
C3	0.1796 (6)	0.0765 (5)	0.51820 (10)	0.1315 (17)
H3	0.2287	0.0428	0.5371	0.158*
C4	0.0693 (7)	0.1363 (6)	0.52598 (13)	0.160 (3)
H4	0.0429	0.1420	0.5504	0.192*
C5	−0.0030 (5)	0.1882 (4)	0.49849 (14)	0.1293 (17)
H5	−0.0779	0.2292	0.5041	0.155*
C6	0.0363 (3)	0.1790 (3)	0.46230 (8)	0.0840 (8)
C7	0.1943 (2)	0.1069 (2)	0.41385 (6)	0.0577 (5)
H7	0.2612	0.0409	0.4137	0.069*
C8	0.2899 (2)	0.2203 (2)	0.36306 (6)	0.0545 (5)
H8	0.3082	0.3110	0.3545	0.065*
C9	0.4104 (2)	0.1421 (2)	0.36226 (6)	0.0564 (6)
C10	0.5121 (3)	0.1931 (3)	0.38075 (8)	0.0844 (8)
H10	0.5061	0.2749	0.3928	0.101*
C11	0.6226 (3)	0.1236 (5)	0.38154 (10)	0.1080 (11)
H11	0.6904	0.1590	0.3940	0.130*
C12	0.6325 (3)	0.0028 (4)	0.36396 (11)	0.1008 (11)
H12	0.7066	−0.0443	0.3649	0.121*
C13	0.5331 (2)	−0.0489 (3)	0.34496 (8)	0.0822 (8)
H13	0.5399	−0.1302	0.3327	0.099*
C14	0.4232 (2)	0.0211 (2)	0.34435 (6)	0.0625 (6)
H14	0.3560	−0.0140	0.3315	0.075*
C15	0.18997 (19)	0.1652 (2)	0.33852 (6)	0.0547 (5)
C16	0.0986 (2)	0.0886 (2)	0.35411 (7)	0.0610 (6)
C17	−0.0003 (2)	0.0385 (3)	0.33265 (9)	0.0773 (7)
H17	−0.0624	−0.0114	0.3438	0.093*

C18	−0.0047 (3)	0.0632 (3)	0.29574 (9)	0.0852 (8)
H18	−0.0697	0.0289	0.2818	0.102*
C19	0.0876 (2)	0.1399 (3)	0.27812 (8)	0.0737 (7)
C20	0.0857 (3)	0.1656 (3)	0.23973 (9)	0.0939 (10)
H20	0.0224	0.1297	0.2254	0.113*
C21	0.1733 (4)	0.2410 (4)	0.22337 (9)	0.1035 (11)
H21	0.1708	0.2556	0.1980	0.124*
C22	0.2678 (3)	0.2969 (3)	0.24464 (7)	0.0895 (9)
H22	0.3269	0.3509	0.2334	0.107*
C23	0.2741 (3)	0.2731 (3)	0.28176 (7)	0.0729 (7)
H23	0.3383	0.3104	0.2955	0.088*
C24	0.1852 (2)	0.1931 (2)	0.29982 (6)	0.0609 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1270 (8)	0.0978 (6)	0.1654 (9)	0.0119 (5)	−0.0736 (7)	−0.0025 (6)
Cl2	0.0843 (6)	0.1073 (7)	0.1950 (11)	0.0303 (5)	0.0179 (6)	0.0009 (7)
O1	0.0700 (11)	0.0685 (10)	0.0757 (11)	−0.0240 (8)	0.0004 (8)	−0.0048 (8)
N1	0.0622 (11)	0.0528 (11)	0.0580 (11)	−0.0092 (9)	0.0081 (9)	−0.0083 (9)
C1	0.0713 (15)	0.0489 (12)	0.0646 (14)	−0.0111 (11)	0.0114 (12)	−0.0030 (10)
C2	0.119 (2)	0.0648 (16)	0.0710 (18)	−0.0322 (16)	−0.0129 (16)	0.0017 (13)
C3	0.210 (5)	0.119 (3)	0.065 (2)	−0.084 (3)	−0.012 (3)	0.002 (2)
C4	0.269 (8)	0.130 (4)	0.082 (3)	−0.111 (5)	0.069 (4)	−0.040 (3)
C5	0.159 (4)	0.091 (3)	0.138 (3)	−0.045 (2)	0.091 (3)	−0.044 (3)
C6	0.090 (2)	0.0641 (15)	0.098 (2)	−0.0122 (14)	0.0370 (17)	−0.0110 (14)
C7	0.0571 (12)	0.0524 (12)	0.0635 (14)	−0.0051 (10)	0.0047 (11)	−0.0061 (10)
C8	0.0579 (13)	0.0556 (13)	0.0499 (12)	−0.0091 (10)	0.0031 (10)	−0.0052 (10)
C9	0.0503 (12)	0.0688 (14)	0.0501 (12)	−0.0107 (11)	0.0028 (10)	0.0029 (10)
C10	0.0659 (17)	0.105 (2)	0.0820 (18)	−0.0120 (16)	−0.0122 (14)	−0.0117 (16)
C11	0.0606 (19)	0.146 (3)	0.118 (3)	−0.018 (2)	−0.0249 (17)	0.009 (2)
C12	0.0507 (16)	0.124 (3)	0.128 (3)	0.0081 (18)	0.0072 (17)	0.032 (2)
C13	0.0639 (17)	0.0863 (19)	0.097 (2)	0.0076 (14)	0.0183 (15)	0.0106 (16)
C14	0.0507 (13)	0.0715 (15)	0.0654 (14)	−0.0034 (11)	0.0071 (10)	−0.0002 (12)
C15	0.0493 (12)	0.0532 (12)	0.0617 (14)	0.0032 (10)	−0.0030 (10)	−0.0102 (10)
C16	0.0564 (13)	0.0573 (13)	0.0693 (16)	−0.0006 (11)	−0.0041 (11)	−0.0107 (11)
C17	0.0573 (14)	0.0706 (16)	0.104 (2)	−0.0080 (12)	−0.0128 (14)	−0.0130 (15)
C18	0.0732 (18)	0.0804 (18)	0.102 (2)	0.0057 (15)	−0.0364 (16)	−0.0224 (16)
C19	0.0710 (16)	0.0706 (16)	0.0795 (18)	0.0176 (14)	−0.0211 (14)	−0.0187 (13)
C20	0.103 (2)	0.102 (2)	0.076 (2)	0.030 (2)	−0.0342 (18)	−0.0223 (17)
C21	0.128 (3)	0.122 (3)	0.0605 (18)	0.041 (2)	−0.016 (2)	−0.0045 (18)
C22	0.104 (2)	0.102 (2)	0.0623 (17)	0.0224 (18)	0.0041 (16)	0.0032 (15)
C23	0.0777 (17)	0.0803 (17)	0.0608 (15)	0.0121 (14)	0.0002 (13)	−0.0053 (13)
C24	0.0617 (14)	0.0614 (13)	0.0594 (14)	0.0156 (12)	−0.0084 (11)	−0.0092 (11)

Geometric parameters (Å, °)

Cl1—C2	1.729 (4)	C11—C12	1.371 (5)
Cl2—C6	1.728 (3)	C11—H11	0.9300

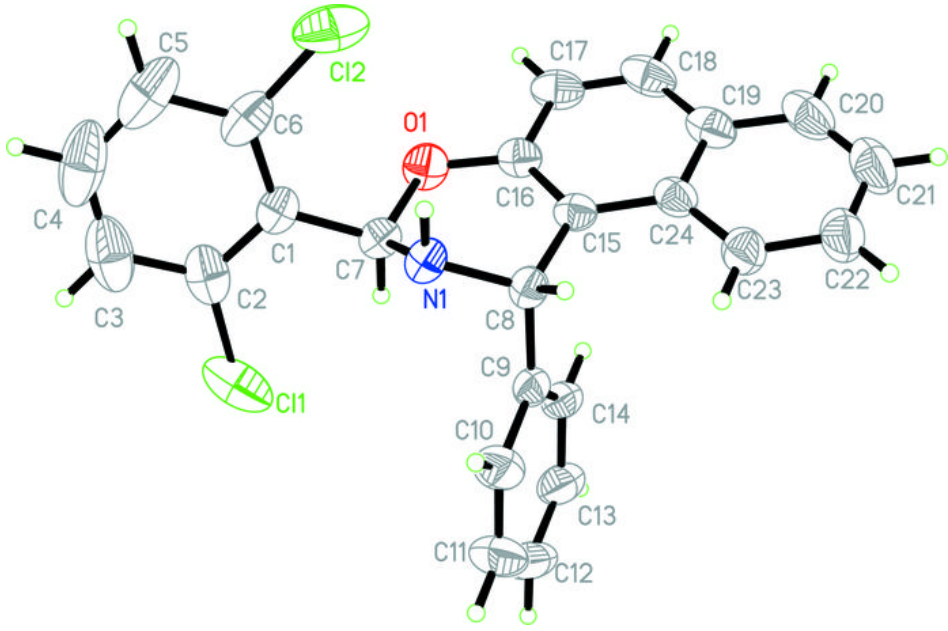
O1—C16	1.371 (3)	C12—C13	1.376 (4)
O1—C7	1.454 (3)	C12—H12	0.9300
N1—C7	1.428 (3)	C13—C14	1.379 (3)
N1—C8	1.476 (3)	C13—H13	0.9300
N1—H1	0.851 (10)	C14—H14	0.9300
C1—C2	1.382 (4)	C15—C16	1.372 (3)
C1—C6	1.397 (4)	C15—C24	1.426 (3)
C1—C7	1.500 (3)	C16—C17	1.412 (3)
C2—C3	1.378 (5)	C17—C18	1.357 (4)
C3—C4	1.363 (7)	C17—H17	0.9300
C3—H3	0.9300	C18—C19	1.410 (4)
C4—C5	1.366 (7)	C18—H18	0.9300
C4—H4	0.9300	C19—C20	1.411 (4)
C5—C6	1.377 (5)	C19—C24	1.418 (3)
C5—H5	0.9300	C20—C21	1.347 (5)
C7—H7	0.9800	C20—H20	0.9300
C8—C15	1.502 (3)	C21—C22	1.396 (5)
C8—C9	1.520 (3)	C21—H21	0.9300
C8—H8	0.9800	C22—C23	1.363 (4)
C9—C14	1.381 (3)	C22—H22	0.9300
C9—C10	1.384 (3)	C23—C24	1.411 (4)
C10—C11	1.382 (5)	C23—H23	0.9300
C10—H10	0.9300		
C16—O1—C7	116.25 (17)	C12—C11—H11	119.9
C7—N1—C8	110.97 (16)	C10—C11—H11	119.9
C7—N1—H1	112.4 (17)	C11—C12—C13	120.2 (3)
C8—N1—H1	108.3 (17)	C11—C12—H12	119.9
C2—C1—C6	116.5 (2)	C13—C12—H12	119.9
C2—C1—C7	120.3 (2)	C12—C13—C14	119.2 (3)
C6—C1—C7	123.2 (2)	C12—C13—H13	120.4
C3—C2—C1	122.3 (4)	C14—C13—H13	120.4
C3—C2—Cl1	117.6 (3)	C13—C14—C9	121.7 (2)
C1—C2—Cl1	120.1 (2)	C13—C14—H14	119.2
C4—C3—C2	119.1 (5)	C9—C14—H14	119.2
C4—C3—H3	120.5	C16—C15—C24	119.1 (2)
C2—C3—H3	120.5	C16—C15—C8	118.7 (2)
C3—C4—C5	121.2 (4)	C24—C15—C8	122.2 (2)
C3—C4—H4	119.4	O1—C16—C15	123.9 (2)
C5—C4—H4	119.4	O1—C16—C17	114.9 (2)
C4—C5—C6	119.2 (5)	C15—C16—C17	121.2 (2)
C4—C5—H5	120.4	C18—C17—C16	120.1 (3)
C6—C5—H5	120.4	C18—C17—H17	120.0
C5—C6—C1	121.8 (4)	C16—C17—H17	120.0
C5—C6—Cl2	117.1 (3)	C17—C18—C19	121.2 (2)
C1—C6—Cl2	121.1 (2)	C17—C18—H18	119.4
N1—C7—O1	114.88 (18)	C19—C18—H18	119.4
N1—C7—C1	111.32 (17)	C18—C19—C20	122.2 (3)
O1—C7—C1	108.22 (17)	C18—C19—C24	118.8 (2)
N1—C7—H7	107.4	C20—C19—C24	119.1 (3)

O1—C7—H7	107.4	C21—C20—C19	121.5 (3)
C1—C7—H7	107.4	C21—C20—H20	119.2
N1—C8—C15	110.36 (17)	C19—C20—H20	119.2
N1—C8—C9	109.43 (17)	C20—C21—C22	119.9 (3)
C15—C8—C9	114.52 (17)	C20—C21—H21	120.1
N1—C8—H8	107.4	C22—C21—H21	120.1
C15—C8—H8	107.4	C23—C22—C21	120.5 (3)
C9—C8—H8	107.4	C23—C22—H22	119.7
C14—C9—C10	118.1 (2)	C21—C22—H22	119.8
C14—C9—C8	123.17 (19)	C22—C23—C24	121.3 (3)
C10—C9—C8	118.7 (2)	C22—C23—H23	119.3
C11—C10—C9	120.7 (3)	C24—C23—H23	119.3
C11—C10—H10	119.7	C23—C24—C19	117.7 (2)
C9—C10—H10	119.7	C23—C24—C15	122.6 (2)
C12—C11—C10	120.1 (3)	C19—C24—C15	119.7 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl2	0.851 (10)	2.88 (2)	3.406 (2)	121.6 (19)

Fig. 1


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