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

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

Key Laboratory of Surface and Interface Science of Henan, School of Materials 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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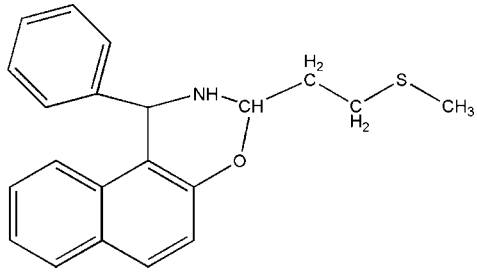
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.122; data-to-parameter ratio = 16.5.

In the title compound, $C_{21}H_{21}NOS$, the oxazine ring is not planar and displays a half-chair conformation. The phenyl and naphthalene ring systems make a dihedral angle of 78.65 (7)°.

Related literature

For related literature, see: Katayama & Oshiyama (1997); Mahajan *et al.* (1991); Mishra *et al.* (1998); Cremer & Pople (1975).



Experimental

Crystal data

$C_{21}H_{21}NOS$
M_r = 335.45
Monoclinic, $P2_1/c$
a = 7.2753 (7) Å
b = 14.9201 (13) Å
c = 16.4212 (15) Å
β = 98.375 (1)°
V = 1763.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 298 (2) K
0.27 × 0.23 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.951, T_{max} = 0.959
20758 measured reflections
3655 independent reflections
2876 reflections with $I > 2/s(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.122
S = 1.05
3655 reflections
221 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); 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: DN2249).

References

Bruker (1998). *SMART* (Version 5.628), *SAINT* (Version 6.02) and *SADABS* (Version 2.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Katayama, H. & Oshiyama, T. (1997). *Can. J. Chem.* **75**, 913–919.
Mahajan, R. N., Havaldar, F. H. & Femandes, P. S. (1991). *J. Indian Chem. Soc.* **68**, 245–249.
Mishra, P. D., Wahidullah, S. & Kamat, S. Y. (1998). *Indian J. Chem. Sect. B*, **37**, 199–200.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

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supplementary materials

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

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Comment

The oxazine derivatives present various applications and widespread potential biological and pharmacological activities such as antimicrobial(Mahajan *et al.*, 1991), antitumor(Katayama & Oshiyama, 1997) and antihistaminic (Mishra *et al.*, 1998). In view of these important properties, the crystal structure of the title compound, (I), has been determined.

The oxazine ring is not planar and displays an half-chair conformation with the puckering parameters: Q= 0.445 (2) Å, θ = 52.9 (2)° and φ= 268.0 (2) °(Cremer & Pople, 1975). The dihedral angle between the benzene ring and the naphthalene ring is 78.65 (7). The bond lengths C18—N1 and C18—O1 in the oxazine ring are 1.421 (2)Å and 1.458 (2)Å respectively.

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, 3-(methylthio)propanal (1 mmol 0.104 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 dichloromethane, colorless single crystals of (I) was obtained after 5 d.

Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic), 0.98 Å (methine), 0.96Å (methyl) and 0.97Å (methylene) with U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(Cmethyl). H atom atom attached to N was 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 represented as small spheres of arbitrary radii.

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

Crystal data

$C_{21}H_{21}NOS$ F_{000} = 712

M_r = 335.45	D_x = 1.263 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1680 reflections
a = 7.2753 (7) Å	θ = 2.5–24.2º
b = 14.9201 (13) Å	μ = 0.19 mm^{-1}
c = 16.4212 (15) Å	T = 298 (2) K
β = 98.3750 (10)º	Block, colorless
V = 1763.5 (3) Å^3	0.27 × 0.23 × 0.23 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3655 independent reflections
Radiation source: fine-focus sealed tube	2876 reflections with $I > 2/s(I)$
Monochromator: graphite	R_{int} = 0.027
T = 298(2) K	θ_{max} = 26.5º
ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	h = −9→9
T_{min} = 0.951, T_{max} = 0.959	k = −18→18
20758 measured reflections	l = −20→20

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.042	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.122	$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 0.3947P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
3655 reflections	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
221 parameters	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
S1	0.14238 (7)	0.88480 (4)	0.31146 (3)	0.06027 (18)
O1	0.25196 (16)	0.89873 (8)	0.05231 (7)	0.0504 (3)
N1	0.58022 (19)	0.87665 (9)	0.08898 (8)	0.0436 (3)
H1	0.599 (3)	0.9329 (7)	0.0971 (12)	0.065*
C1	0.4372 (2)	0.87861 (9)	−0.05684 (9)	0.0386 (3)
C2	0.2738 (2)	0.90140 (10)	−0.02920 (10)	0.0418 (4)
C3	0.1183 (2)	0.93206 (12)	−0.08314 (11)	0.0511 (4)
H3	0.0104	0.9479	−0.0625	0.061*
C4	0.1246 (2)	0.93857 (12)	−0.16469 (11)	0.0539 (4)
H4	0.0210	0.9590	−0.1997	0.065*
C5	0.2875 (3)	0.91463 (11)	−0.19755 (10)	0.0478 (4)
C6	0.2985 (3)	0.92130 (12)	−0.28267 (11)	0.0602 (5)
H6	0.1968	0.9428	−0.3182	0.072*
C7	0.4539 (4)	0.89713 (13)	−0.31338 (11)	0.0663 (6)
H7	0.4576	0.9009	−0.3696	0.080*
C8	0.6092 (3)	0.86646 (13)	−0.26024 (12)	0.0624 (5)
H8	0.7159	0.8503	−0.2816	0.075*
C9	0.6063 (3)	0.85996 (12)	−0.17763 (11)	0.0510 (4)
H9	0.7111	0.8396	−0.1434	0.061*
C10	0.4449 (2)	0.88390 (9)	−0.14325 (9)	0.0405 (3)
C11	0.6056 (2)	0.85344 (10)	0.00404 (9)	0.0402 (3)
H11	0.7098	0.8893	−0.0095	0.048*
C12	0.6629 (2)	0.75561 (11)	0.00149 (9)	0.0424 (4)
C13	0.5476 (2)	0.69028 (11)	−0.03684 (10)	0.0487 (4)
H13	0.4308	0.7061	−0.0639	0.058*
C14	0.6025 (3)	0.60134 (12)	−0.03570 (13)	0.0643 (5)
H14	0.5230	0.5582	−0.0621	0.077*
C15	0.7718 (4)	0.57695 (16)	0.00378 (14)	0.0783 (7)
H15	0.8073	0.5170	0.0057	0.094*
C16	0.8897 (4)	0.64095 (19)	0.04070 (16)	0.0933 (9)
H16	1.0070	0.6245	0.0667	0.112*
C17	0.8365 (3)	0.73032 (16)	0.03989 (13)	0.0729 (6)
H17	0.9182	0.7733	0.0653	0.087*
C18	0.4003 (2)	0.85325 (11)	0.10595 (9)	0.0440 (4)
H18	0.3838	0.7884	0.0985	0.053*
C19	0.3727 (2)	0.87701 (12)	0.19314 (10)	0.0497 (4)
H19A	0.4702	0.8493	0.2316	0.060*
H19B	0.3826	0.9414	0.2004	0.060*
C20	0.1860 (3)	0.84608 (13)	0.21210 (11)	0.0563 (5)
H20A	0.1815	0.7811	0.2108	0.068*

H20B	0.0893	0.8683	0.1700	0.068*
C21	−0.0719 (4)	0.8270 (2)	0.31290 (18)	0.1074 (10)
H21A	−0.1599	0.8456	0.2666	0.161*
H21B	−0.1196	0.8408	0.3630	0.161*
H21C	−0.0515	0.7636	0.3099	0.161*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0599 (3)	0.0828 (4)	0.0395 (3)	0.0056 (2)	0.0120 (2)	−0.0013 (2)
O1	0.0468 (6)	0.0636 (7)	0.0432 (6)	0.0048 (5)	0.0144 (5)	0.0004 (5)
N1	0.0464 (8)	0.0453 (7)	0.0394 (7)	−0.0052 (6)	0.0077 (6)	−0.0083 (6)
C1	0.0437 (8)	0.0321 (7)	0.0406 (8)	−0.0016 (6)	0.0084 (6)	−0.0004 (6)
C2	0.0453 (9)	0.0400 (8)	0.0409 (8)	−0.0017 (7)	0.0093 (7)	−0.0014 (6)
C3	0.0421 (9)	0.0542 (10)	0.0571 (10)	0.0030 (7)	0.0076 (8)	−0.0020 (8)
C4	0.0503 (10)	0.0517 (10)	0.0559 (10)	0.0028 (8)	−0.0052 (8)	0.0006 (8)
C5	0.0609 (10)	0.0382 (8)	0.0428 (9)	−0.0040 (7)	0.0025 (7)	−0.0008 (7)
C6	0.0847 (14)	0.0532 (10)	0.0393 (9)	−0.0036 (10)	−0.0025 (9)	0.0021 (8)
C7	0.1079 (17)	0.0558 (11)	0.0370 (9)	−0.0052 (11)	0.0160 (10)	−0.0007 (8)
C8	0.0847 (14)	0.0576 (11)	0.0508 (11)	0.0023 (10)	0.0295 (10)	−0.0011 (8)
C9	0.0600 (11)	0.0483 (9)	0.0473 (9)	0.0025 (8)	0.0167 (8)	0.0011 (7)
C10	0.0507 (9)	0.0324 (7)	0.0392 (8)	−0.0028 (6)	0.0087 (7)	−0.0014 (6)
C11	0.0405 (8)	0.0431 (8)	0.0377 (8)	−0.0040 (6)	0.0079 (6)	−0.0028 (6)
C12	0.0455 (8)	0.0491 (9)	0.0332 (7)	0.0069 (7)	0.0082 (6)	0.0008 (6)
C13	0.0508 (9)	0.0431 (9)	0.0530 (10)	0.0024 (7)	0.0105 (8)	0.0024 (7)
C14	0.0826 (15)	0.0451 (10)	0.0696 (13)	0.0042 (9)	0.0255 (11)	0.0011 (9)
C15	0.1074 (19)	0.0586 (12)	0.0715 (14)	0.0345 (13)	0.0214 (13)	0.0102 (11)
C16	0.0943 (19)	0.102 (2)	0.0754 (15)	0.0557 (16)	−0.0145 (13)	−0.0026 (14)
C17	0.0623 (12)	0.0832 (15)	0.0656 (12)	0.0228 (11)	−0.0164 (10)	−0.0176 (11)
C18	0.0497 (9)	0.0422 (8)	0.0405 (8)	−0.0015 (7)	0.0081 (7)	−0.0018 (6)
C19	0.0557 (10)	0.0533 (10)	0.0413 (9)	−0.0024 (8)	0.0110 (8)	−0.0036 (7)
C20	0.0648 (11)	0.0593 (11)	0.0477 (10)	−0.0080 (9)	0.0184 (8)	−0.0074 (8)
C21	0.099 (2)	0.131 (2)	0.106 (2)	−0.0306 (18)	0.0601 (17)	−0.0140 (18)

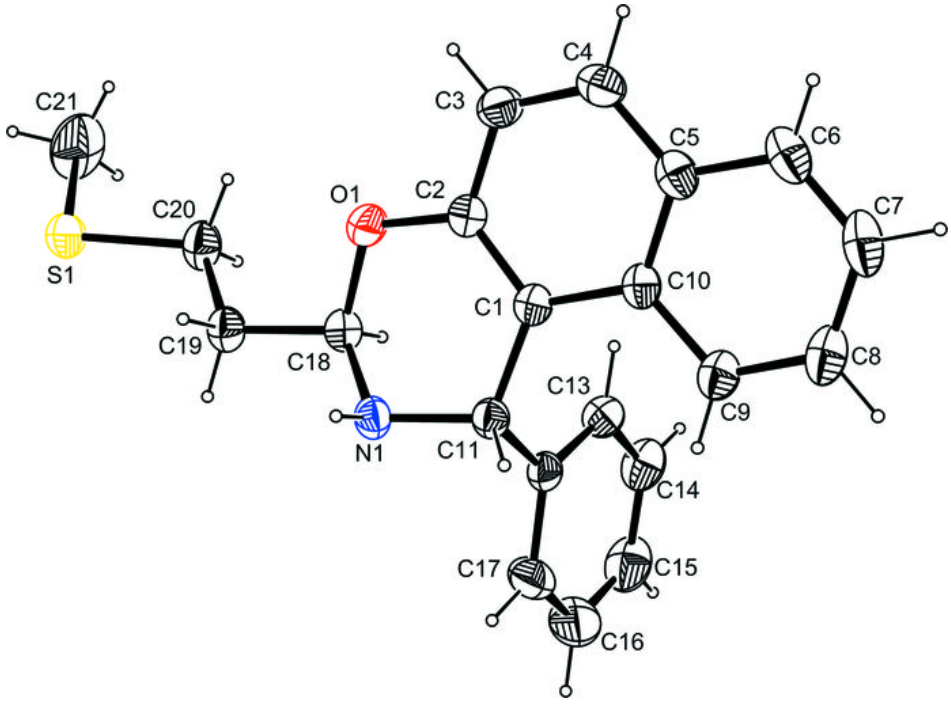
Geometric parameters (Å, °)

S1—C21	1.784 (3)	C9—H9	0.9300
S1—C20	1.8020 (17)	C11—C12	1.520 (2)
O1—C2	1.3714 (19)	C11—H11	0.9800
O1—C18	1.457 (2)	C12—C13	1.377 (2)
N1—C18	1.421 (2)	C12—C17	1.380 (2)
N1—C11	1.4745 (19)	C13—C14	1.385 (2)
N1—H1	0.857 (9)	C13—H13	0.9300
C1—C2	1.375 (2)	C14—C15	1.355 (3)
C1—C10	1.430 (2)	C14—H14	0.9300
C1—C11	1.511 (2)	C15—C16	1.365 (4)
C2—C3	1.408 (2)	C15—H15	0.9300
C3—C4	1.350 (2)	C16—C17	1.388 (3)
C3—H3	0.9300	C16—H16	0.9300

C4—C5	1.417 (3)	C17—H17	0.9300
C4—H4	0.9300	C18—C19	1.516 (2)
C5—C6	1.415 (2)	C18—H18	0.9800
C5—C10	1.421 (2)	C19—C20	1.510 (2)
C6—C7	1.352 (3)	C19—H19A	0.9700
C6—H6	0.9300	C19—H19B	0.9700
C7—C8	1.400 (3)	C20—H20A	0.9700
C7—H7	0.9300	C20—H20B	0.9700
C8—C9	1.363 (2)	C21—H21A	0.9600
C8—H8	0.9300	C21—H21B	0.9600
C9—C10	1.421 (2)	C21—H21C	0.9600
C21—S1—C20	97.27 (11)	C13—C12—C11	122.72 (14)
C2—O1—C18	115.00 (12)	C17—C12—C11	119.12 (16)
C18—N1—C11	111.93 (12)	C12—C13—C14	121.08 (18)
C18—N1—H1	109.7 (14)	C12—C13—H13	119.5
C11—N1—H1	109.8 (13)	C14—C13—H13	119.5
C2—C1—C10	118.30 (14)	C15—C14—C13	120.3 (2)
C2—C1—C11	119.96 (13)	C15—C14—H14	119.9
C10—C1—C11	121.67 (13)	C13—C14—H14	119.9
O1—C2—C1	122.92 (14)	C14—C15—C16	119.5 (2)
O1—C2—C3	115.26 (14)	C14—C15—H15	120.2
C1—C2—C3	121.79 (15)	C16—C15—H15	120.2
C4—C3—C2	120.46 (16)	C15—C16—C17	120.8 (2)
C4—C3—H3	119.8	C15—C16—H16	119.6
C2—C3—H3	119.8	C17—C16—H16	119.6
C3—C4—C5	120.71 (16)	C12—C17—C16	120.2 (2)
C3—C4—H4	119.6	C12—C17—H17	119.9
C5—C4—H4	119.6	C16—C17—H17	119.9
C6—C5—C4	121.93 (17)	N1—C18—O1	112.90 (13)
C6—C5—C10	119.11 (17)	N1—C18—C19	112.33 (13)
C4—C5—C10	118.96 (15)	O1—C18—C19	105.88 (13)
C7—C6—C5	121.34 (19)	N1—C18—H18	108.5
C7—C6—H6	119.3	O1—C18—H18	108.5
C5—C6—H6	119.3	C19—C18—H18	108.5
C6—C7—C8	119.82 (17)	C20—C19—C18	111.72 (14)
C6—C7—H7	120.1	C20—C19—H19A	109.3
C8—C7—H7	120.1	C18—C19—H19A	109.3
C9—C8—C7	121.03 (19)	C20—C19—H19B	109.3
C9—C8—H8	119.5	C18—C19—H19B	109.3
C7—C8—H8	119.5	H19A—C19—H19B	107.9
C8—C9—C10	120.73 (18)	C19—C20—S1	111.79 (12)
C8—C9—H9	119.6	C19—C20—H20A	109.3
C10—C9—H9	119.6	S1—C20—H20A	109.3
C9—C10—C5	117.96 (15)	C19—C20—H20B	109.3
C9—C10—C1	122.29 (15)	S1—C20—H20B	109.3
C5—C10—C1	119.75 (14)	H20A—C20—H20B	107.9
N1—C11—C1	111.50 (12)	S1—C21—H21A	109.5
N1—C11—C12	108.91 (12)	S1—C21—H21B	109.5
C1—C11—C12	114.81 (13)	H21A—C21—H21B	109.5

N1—C11—H11	107.1	S1—C21—H21C	109.5
C1—C11—H11	107.1	H21A—C21—H21C	109.5
C12—C11—H11	107.1	H21B—C21—H21C	109.5
C13—C12—C17	118.15 (17)		

Fig. 1


C21
C4
C3
C5
C6
C7
O1
C2
C20
S1
C10
C1
C19
C18
C8
C13
C9
N1
C11
C14
C17
C15
C16