

2,6-Bis[(4*R*,5*R*)-4,5-diphenyl-4,5-dihydro-1,3-oxazol-2-yl]pyridine

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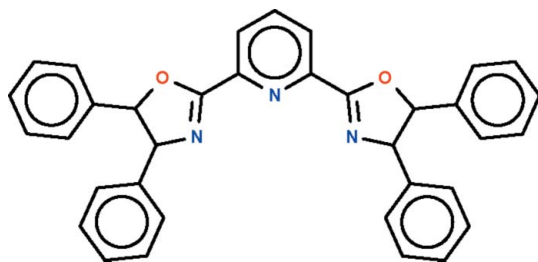
Received 17 April 2011; accepted 19 April 2011

Key indicators: single-crystal X-ray study; $T = 293\text{ K}$; mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$; R factor = 0.034; wR factor = 0.099; data-to-parameter ratio = 7.1.

The molecule of the title compound, $\text{C}_{35}\text{H}_{27}\text{N}_3\text{O}_2$, lies on a twofold rotation axis passing through the pyridine ring. The five-membered ring is approximately flat (r.m.s. deviation = $0.065\text{ \AA}$) and is essentially coplanar [dihedral angle = $4.2(2)^\circ$] with the pyridine ring.

Related literature

For the synthesis of the precursor, see: Desimoni *et al.* (2001). For the structure of 2,6-bis(2-oxazolinyl)pyridine, see: Sada *et al.* (2003).



Experimental

Crystal data

$\text{C}_{35}\text{H}_{27}\text{N}_3\text{O}_2$
 $M_r = 521.60$
Monoclinic, $C2$
 $a = 19.035(2)\text{ \AA}$
 $b = 6.5908(7)\text{ \AA}$
 $c = 14.3001(15)\text{ \AA}$
 $\beta = 129.454(1)^\circ$

$V = 1385.2(3)\text{ \AA}^3$
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.08\text{ mm}^{-1}$
 $T = 293\text{ K}$
 $0.38 \times 0.20 \times 0.16\text{ mm}$

Data collection

Bruker SMART CCD
diffractometer
3266 measured reflections

1299 independent reflections
1190 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.099$
 $S = 1.05$
1299 reflections
182 parameters
1 restraint

H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.16\text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.11\text{ e \AA}^{-3}$
Absolute structure: 768 Friedel
pairs merged

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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, 2010).

The authors thank the Scientific Research Foundation for Returned Overseas Chinese Scholars and the Programme for New Century Excellent Talents (both from the State Education Ministry of China), the Project of International Science and Technology Cooperation (from the Ministry of Science and Technology of China) and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5522).

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supporting information

Acta Cryst. (2011). E67, o1344 [<https://doi.org/10.1107/S1600536811014668>]

2,6-Bis[(4*R*,5*R*)-4,5-diphenyl-4,5-dihydro-1,3-oxazol-2-yl]pyridine

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S1. Comment

We report here the cyclization of the two side arms of (2,6-bis[(1*R*,2*S*)*N,N'*-2-chloro-1,2-diphenylethyl]-pyridine-dicarboxamide to form a di-substituted pyridine having two 1,3-oxazoline rings. The title compound is intended for an evaluation of its pharmaceutical properties. It lies on a twofold rotation axis that passes through the pyridine ring (Fig. 1). The five-membered ring is approximately flat [r.m.s. deviation 0.065 Å] and is nearly coplanar [dihedral angle 4.2 (2)°] with the pyridine ring. In the parent compound, 2,6-bis(2-oxazoliny)pyridine, the three rings are similarly nearly coplanar (Sada *et al.*, 2003).

S2. Experimental

To a suspension of 2,6-bis[(1*R*,2*S*)*N,N'*-2-chloro-1,2-diphenylethyl]-pyridinedicarboxamide (Desimoni *et al.*, 2001) (0.89 g, 1.5 mmol) in ethanol (26 ml), an aqueous solution of 2 N sodium hydroxide (13 ml) was added and the mixture was refluxed 8 h. The hot suspension was filtered and the white solid was washed with water (0.73 g, yield 90%). Crystals were obtained by recrystallization from ethyl acetate; m.p. 474–474 K.

S3. Refinement

H atoms were placed in calculated positions (C—H 0.93–0.99 Å) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$; 768 Friedel pairs were merged. The absolute configuration was assumed to be that of the reactant.

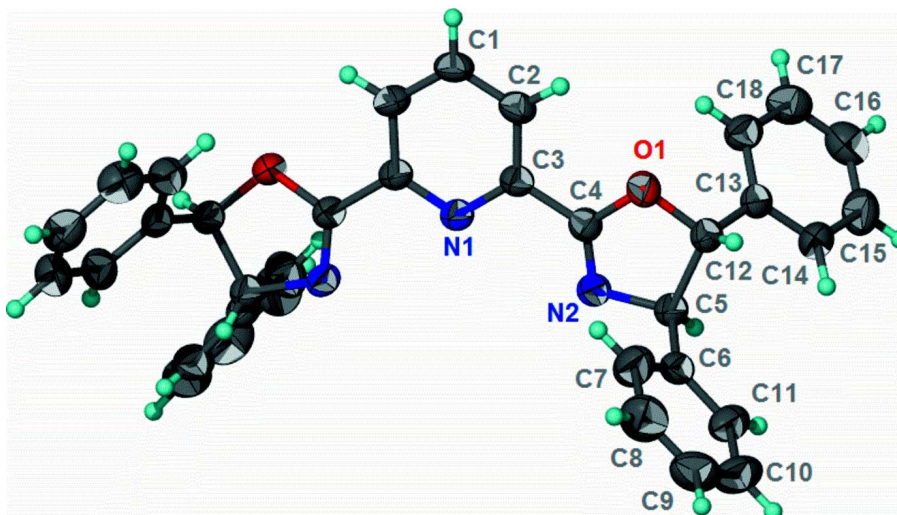


Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{35}H_{27}N_3O_2$ at the 50% probability level; H atoms are drawn as arbitrary radius.

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Crystal data

$C_{35}H_{27}N_3O_2$
 $M_r = 521.60$
 Monoclinic, $C2$
 Hall symbol: $C\ 2y$
 $a = 19.035\ (2)\ \text{\AA}$
 $b = 6.5908\ (7)\ \text{\AA}$
 $c = 14.3001\ (15)\ \text{\AA}$
 $\beta = 129.454\ (1)^\circ$
 $V = 1385.2\ (3)\ \text{\AA}^3$
 $Z = 2$

$F(000) = 548$
 $D_x = 1.251\ \text{Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$
 Cell parameters from 1652 reflections
 $\theta = 2.8\text{--}26.1^\circ$
 $\mu = 0.08\ \text{mm}^{-1}$
 $T = 293\ \text{K}$
 Prism, colourless
 $0.38 \times 0.20 \times 0.16\ \text{mm}$

Data collection

Bruker SMART CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 3266 measured reflections
 1299 independent reflections

1190 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$
 $\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 2.9^\circ$
 $h = -22 \rightarrow 22$
 $k = -7 \rightarrow 7$
 $l = -17 \rightarrow 15$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.099$
 $S = 1.05$
 1299 reflections
 182 parameters
 1 restraint

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
 $w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 0.0677P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.11 \text{ e } \text{\AA}^{-3}$$

Absolute structure: 768 Friedel pairs merged

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	−0.00290 (12)	0.4999 (2)	0.24429 (14)	0.0590 (5)
N1	0.0000	0.3969 (4)	0.0000	0.0453 (6)
N2	0.00872 (13)	0.1951 (3)	0.18302 (16)	0.0503 (5)
C1	0.0000	0.8221 (5)	0.0000	0.0582 (8)
H1	0.0000	0.9632	0.0000	0.070*
C2	−0.00113 (15)	0.7152 (4)	0.0817 (2)	0.0538 (6)
H2	−0.0023	0.7831	0.1377	0.065*
C3	−0.00049 (13)	0.5041 (3)	0.07933 (18)	0.0449 (5)
C4	0.00168 (14)	0.3864 (4)	0.16940 (19)	0.0472 (5)
C5	0.00709 (15)	0.1499 (4)	0.2830 (2)	0.0505 (5)
H5	0.0604	0.0679	0.3445	0.061*
C6	−0.07812 (16)	0.0392 (4)	0.2397 (2)	0.0522 (5)
C7	−0.16168 (18)	0.0935 (5)	0.1346 (2)	0.0769 (8)
H7	−0.1661	0.1950	0.0859	0.092*
C8	−0.2399 (2)	−0.0029 (6)	0.1005 (3)	0.0909 (10)
H8	−0.2964	0.0356	0.0294	0.109*
C9	−0.2346 (2)	−0.1521 (6)	0.1699 (4)	0.0887 (10)
H9	−0.2872	−0.2154	0.1468	0.106*
C10	−0.1519 (2)	−0.2093 (5)	0.2738 (3)	0.0833 (9)
H10	−0.1481	−0.3124	0.3212	0.100*
C11	−0.07370 (18)	−0.1150 (4)	0.3090 (2)	0.0648 (7)
H11	−0.0175	−0.1553	0.3800	0.078*
C12	0.01565 (15)	0.3640 (4)	0.33649 (19)	0.0498 (5)
H12	−0.0314	0.3795	0.3447	0.060*
C13	0.10798 (15)	0.4089 (4)	0.45661 (19)	0.0493 (5)
C14	0.14229 (19)	0.2821 (5)	0.5543 (2)	0.0654 (7)
H14	0.1074	0.1742	0.5463	0.078*
C15	0.2283 (2)	0.3148 (6)	0.6638 (3)	0.0788 (8)
H15	0.2515	0.2268	0.7284	0.095*
C16	0.27910 (19)	0.4766 (5)	0.6770 (3)	0.0738 (8)
H16	0.3367	0.4995	0.7506	0.089*
C17	0.24501 (16)	0.6032 (5)	0.5823 (3)	0.0712 (8)
H17	0.2794	0.7136	0.5916	0.085*
C18	0.16001 (17)	0.5710 (4)	0.4720 (2)	0.0600 (6)
H18	0.1378	0.6592	0.4079	0.072*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0850 (11)	0.0442 (10)	0.0564 (9)	0.0107 (8)	0.0489 (9)	0.0016 (8)
N1	0.0524 (14)	0.0389 (14)	0.0499 (14)	0.000	0.0350 (12)	0.000
N2	0.0666 (11)	0.0425 (12)	0.0597 (11)	−0.0016 (9)	0.0486 (10)	−0.0038 (9)

C1	0.070 (2)	0.0344 (17)	0.061 (2)	0.000	0.0377 (17)	0.000
C2	0.0642 (14)	0.0395 (13)	0.0570 (13)	0.0013 (11)	0.0381 (12)	−0.0048 (11)
C3	0.0466 (12)	0.0397 (13)	0.0473 (11)	0.0004 (9)	0.0294 (10)	−0.0013 (9)
C4	0.0525 (12)	0.0443 (13)	0.0496 (12)	0.0032 (10)	0.0347 (10)	−0.0006 (10)
C5	0.0618 (13)	0.0449 (13)	0.0549 (12)	−0.0013 (10)	0.0418 (11)	−0.0010 (11)
C6	0.0666 (13)	0.0445 (12)	0.0565 (12)	−0.0065 (11)	0.0442 (12)	−0.0076 (10)
C7	0.0767 (17)	0.079 (2)	0.0661 (15)	−0.0148 (16)	0.0411 (15)	0.0034 (15)
C8	0.0714 (18)	0.103 (3)	0.0825 (19)	−0.0200 (19)	0.0413 (16)	−0.010 (2)
C9	0.090 (2)	0.083 (2)	0.117 (2)	−0.0336 (19)	0.078 (2)	−0.029 (2)
C10	0.106 (2)	0.0622 (18)	0.122 (2)	−0.0105 (17)	0.092 (2)	0.0006 (19)
C11	0.0817 (16)	0.0521 (14)	0.0830 (17)	0.0032 (13)	0.0629 (15)	0.0058 (14)
C12	0.0648 (13)	0.0450 (13)	0.0543 (12)	0.0017 (11)	0.0448 (12)	−0.0005 (10)
C13	0.0642 (13)	0.0468 (13)	0.0546 (12)	−0.0019 (10)	0.0460 (11)	−0.0041 (10)
C14	0.0837 (17)	0.0614 (17)	0.0612 (14)	−0.0122 (14)	0.0507 (14)	−0.0017 (13)
C15	0.0943 (19)	0.080 (2)	0.0591 (15)	0.0159 (19)	0.0474 (15)	0.0108 (16)
C16	0.0644 (15)	0.078 (2)	0.0718 (16)	0.0007 (15)	0.0399 (14)	−0.0115 (16)
C17	0.0644 (16)	0.0689 (18)	0.0885 (18)	−0.0091 (14)	0.0523 (16)	−0.0086 (16)
C18	0.0687 (15)	0.0554 (15)	0.0696 (14)	−0.0040 (13)	0.0504 (13)	0.0000 (12)

Geometric parameters (Å, °)

O1—C4	1.354 (3)	C8—H8	0.9300
O1—C12	1.440 (3)	C9—C10	1.362 (5)
N1—C3	1.341 (2)	C9—H9	0.9300
N1—C3 ⁱ	1.341 (2)	C10—C11	1.379 (4)
N2—C4	1.270 (3)	C10—H10	0.9300
N2—C5	1.480 (3)	C11—H11	0.9300
C1—C2	1.376 (3)	C12—C13	1.513 (3)
C1—C2 ⁱ	1.376 (3)	C12—H12	0.9800
C1—H1	0.9300	C13—C18	1.376 (3)
C2—C3	1.392 (3)	C13—C14	1.384 (3)
C2—H2	0.9300	C14—C15	1.386 (4)
C3—C4	1.481 (3)	C14—H14	0.9300
C5—C6	1.507 (3)	C15—C16	1.370 (5)
C5—C12	1.563 (3)	C15—H15	0.9300
C5—H5	0.9800	C16—C17	1.355 (4)
C6—C7	1.371 (4)	C16—H16	0.9300
C6—C11	1.384 (3)	C17—C18	1.381 (4)
C7—C8	1.392 (4)	C17—H17	0.9300
C7—H7	0.9300	C18—H18	0.9300
C8—C9	1.355 (5)		
C4—O1—C12	106.19 (17)	C10—C9—H9	120.1
C3—N1—C3 ⁱ	116.5 (2)	C9—C10—C11	120.4 (3)
C4—N2—C5	106.44 (19)	C9—C10—H10	119.8
C2—C1—C2 ⁱ	118.4 (3)	C11—C10—H10	119.8
C2—C1—H1	120.8	C10—C11—C6	120.6 (3)
C2 ⁱ —C1—H1	120.8	C10—C11—H11	119.7

C1—C2—C3	119.1 (2)	C6—C11—H11	119.7
C1—C2—H2	120.5	O1—C12—C13	110.46 (18)
C3—C2—H2	120.5	O1—C12—C5	103.00 (15)
N1—C3—C2	123.5 (2)	C13—C12—C5	114.64 (19)
N1—C3—C4	116.65 (18)	O1—C12—H12	109.5
C2—C3—C4	119.9 (2)	C13—C12—H12	109.5
N2—C4—O1	118.7 (2)	C5—C12—H12	109.5
N2—C4—C3	126.5 (2)	C18—C13—C14	118.5 (2)
O1—C4—C3	114.73 (18)	C18—C13—C12	122.2 (2)
N2—C5—C6	112.22 (19)	C14—C13—C12	119.3 (2)
N2—C5—C12	103.37 (18)	C13—C14—C15	120.5 (3)
C6—C5—C12	112.88 (18)	C13—C14—H14	119.7
N2—C5—H5	109.4	C15—C14—H14	119.7
C6—C5—H5	109.4	C16—C15—C14	120.0 (3)
C12—C5—H5	109.4	C16—C15—H15	120.0
C7—C6—C11	118.5 (2)	C14—C15—H15	120.0
C7—C6—C5	121.4 (2)	C17—C16—C15	119.6 (3)
C11—C6—C5	120.1 (2)	C17—C16—H16	120.2
C6—C7—C8	120.2 (3)	C15—C16—H16	120.2
C6—C7—H7	119.9	C16—C17—C18	121.1 (3)
C8—C7—H7	119.9	C16—C17—H17	119.5
C9—C8—C7	120.6 (3)	C18—C17—H17	119.5
C9—C8—H8	119.7	C13—C18—C17	120.3 (3)
C7—C8—H8	119.7	C13—C18—H18	119.9
C8—C9—C10	119.7 (3)	C17—C18—H18	119.9
C8—C9—H9	120.1		
C2 ⁱ —C1—C2—C3	0.42 (15)	C8—C9—C10—C11	0.5 (5)
C3 ⁱ —N1—C3—C2	0.45 (17)	C9—C10—C11—C6	0.1 (5)
C3 ⁱ —N1—C3—C4	−178.3 (2)	C7—C6—C11—C10	−1.0 (4)
C1—C2—C3—N1	−0.9 (3)	C5—C6—C11—C10	176.2 (3)
C1—C2—C3—C4	177.79 (15)	C4—O1—C12—C13	109.2 (2)
C5—N2—C4—O1	1.7 (3)	C4—O1—C12—C5	−13.6 (2)
C5—N2—C4—C3	−179.57 (19)	N2—C5—C12—O1	14.4 (2)
C12—O1—C4—N2	8.5 (3)	C6—C5—C12—O1	−107.09 (19)
C12—O1—C4—C3	−170.44 (17)	N2—C5—C12—C13	−105.65 (19)
N1—C3—C4—N2	3.9 (3)	C6—C5—C12—C13	132.9 (2)
C2—C3—C4—N2	−174.8 (2)	O1—C12—C13—C18	5.3 (3)
N1—C3—C4—O1	−177.25 (15)	C5—C12—C13—C18	121.1 (2)
C2—C3—C4—O1	4.0 (3)	O1—C12—C13—C14	−173.81 (18)
C4—N2—C5—C6	111.8 (2)	C5—C12—C13—C14	−58.0 (3)
C4—N2—C5—C12	−10.1 (3)	C18—C13—C14—C15	−1.9 (4)
N2—C5—C6—C7	−42.1 (3)	C12—C13—C14—C15	177.2 (2)
C12—C5—C6—C7	74.2 (3)	C13—C14—C15—C16	1.7 (4)
N2—C5—C6—C11	140.8 (2)	C14—C15—C16—C17	−0.4 (4)
C12—C5—C6—C11	−102.8 (2)	C15—C16—C17—C18	−0.5 (4)
C11—C6—C7—C8	1.1 (4)	C14—C13—C18—C17	1.0 (3)
C5—C6—C7—C8	−176.0 (3)	C12—C13—C18—C17	−178.2 (2)

C6—C7—C8—C9	−0.5 (5)	C16—C17—C18—C13	0.3 (4)
C7—C8—C9—C10	−0.3 (5)		

Symmetry code: (i) $-x, y, -z$.