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(*R*)-4-Phenyl-2-[(*S*)-1,2,3,4-tetrahydroisoquinolin-3-yl]-4,5-dihydro-1,3-oxazole

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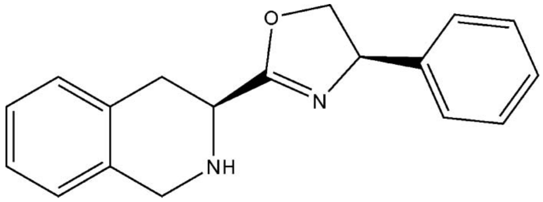
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.076; data-to-parameter ratio = 8.0.

The asymmetric unit cell of the title compound, $C_{18}H_{18}N_2O$, contains four molecules. In the crystal structure, an intermolecular N—H···N hydrogen bond helps to establish the packing.

Related literature

For the assymetric synthetic applications of oxazoline, see: Hargaden *et al.* (2009). For tetraisoquinolines and their biological significance, see: Scott *et al.* (2002). For ligand catalysis activity, see: Chakka *et al.* (2010).



Experimental

Crystal data

$C_{18}H_{18}N_2O$	a = 5.4023 (3) Å
M_r = 278.34	b = 10.0999 (6) Å
Orthorhombic, $P2_12_12_1$	c = 26.2205 (17) Å
V = 1430.66 (15) Å^3	μ = 0.64 mm^{-1}
Z = 4	T = 173 K
Cu $K\alpha$ radiation	0.22 × 0.21 × 0.10 mm

Data collection

Bruker Kappa DUO APEXII diffractometer	6634 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	1552 independent reflections
$T_{\min}$ = 0.682, $T_{\max}$ = 0.753	1479 reflections with $I > 2\sigma(I)$
	$R_{\rm int}$ = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.076	$\Delta\rho_{\max}$ = 0.14 e Å^{-3}
S = 1.05	$\Delta\rho_{\min}$ = −0.12 e Å^{-3}
1552 reflections	
195 parameters	
1 restraint	

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···N2$^{\rm i}$	0.96 (1)	2.20 (1)	3.139 (2)	165 (2)

Symmetry code: (i) $x + 1, y, z$.

Data collection: *SAINT* (Bruker, 2006); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; 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: *SHELXL97*.

The authors would like to thank Dr Hong Su (University of Cape Town) for the data collection and structure refinement.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2694).

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supplementary materials

Acta Cryst. (2010). E**66**, o1818 [**doi:10.1107/S1600536810022130**]

(*R*)-4-Phenyl-2-[(*S*)-1,2,3,4-tetrahydroisoquinolin-3-yl]-4,5-dihydro-1,3-oxazole

S. K. Chakka, T. Govender, H. G. Kruger and G. E. M. Maguire

Comment

Heterocyclic rings play roles in a number of key areas of organic and inorganic chemistry. As part of an ongoing study into the asymmetric hydrogen transfer reactions we made a series of ligands (Chakka *et al.*, 2010). The title compound (**I**) is one such molecule (Fig 1). It combines for the first time two important heterocyclic rings into a single structure, namely oxazoline (Hargaden *et al.*, 2009) and tetraisoquinoline (TIQ), (Scott *et al.*, 2002). The asymmetric unit cell contains four molecules. There are inter-molecular N(1)—H···N(1) bond interactions (2.212 Å) that hold the structure in two dimensional planes. The intermolecular distance value between ring centroids in the *b* axis direction (5.402 Å), suggests that there is no π-stacking interaction between parallel molecules (Fig 2).

Experimental

A solution of Cbz-protected TIQ–oxazoline (1.0 g, 2.89 mmol) in methanol (30 ml) was added to a suspension of 10 wt.% Pd/C (0.5 g) in methanol (10 ml). The reaction mixture was connected to an H_2 source at atmospheric pressure and stirred at room temperature for 3 h. Completion of the reaction was monitored by TLC using hexane/ethyl acetate (7:3) with the R_f = 0.6. The Pd/C was filtered off over a celite pad and the filtrate was concentrated under reduced pressure to afford crude (**I**). The title compound was purified on a deactivated silica gel column packed with a suspension of silica gel in 20% Et_3N/CH_2Cl_2. The silica was washed with 1% Et_3N/CH_2Cl_2. The chromatography was then performed using 0–2% MeOH/1%Et_3N/CH_2Cl_2 as the eluent to afford TIQ-oxazoline product. *M*.p: 304 – 306 K.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37–7.23 (m, 3H), 7.23–7.10 (m, 5H), 7.05 (m, 1H), 5.23 (t, *J* = 18.21 Hz, 1H), 4.68 (q, *J* = 10.10, 8.26 Hz, 1H), 4.16 (m, 3H), 3.92 (m, 1H), 3.10–3.05 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.9, 142.0, 135.0, 133.3, 129.2, 128.7, 127.6, 126.5, 126.2, 126.1, 126.0, 69.4, 51.5, 47.6, 32.4.

IR (neat): 3225, 1663, 1493, 1108, 957, 907, 916, 740, 749, 697 cm^{-1}.

HR ESI MS: 279.1492 $[M + H]^+$ (calcd. for $C_{18}H_{19}N_2O$ 279.1512).

Refinement

All hydrogen atoms, except H1N on N1, were positioned geometrically with C—H = 0.95 - 1.00 Å and refined as riding on their parent atoms with U_{iso} (H) = 1.2 - 1.5 U_{eq} (C). The hydrogen atoms H1N were located in the difference electron density maps and refined with simple bond length constraint.

Figures

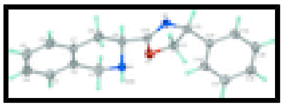

Fig. 1. Molecular structure of (**I**) showing numbering scheme. All non-hydrogen atoms are shown as ellipsoids with probability level of 50%.

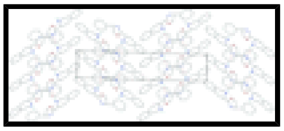

Fig. 2. Projection viewed along [010]. All hydrogen atoms except the hydrogen H1N on N1 are omitted. The hydrogen bonds are shown as dotted lines.

(*R*)-4-Phenyl-2-[(*S*)-1,2,3,4-tetrahydroisoquinolin-3-yl]-4,5- dihydro-1,3-oxazole

Crystal data

$C_{18}H_{18}N_2O$

$M_r = 278.34$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 5.4023$ (3) Å

$b = 10.0999$ (6) Å

$c = 26.2205$ (17) Å

$V = 1430.66$ (15) Å^3

$Z = 4$

$F(000) = 592$

$D_x = 1.292$ Mg m^{-3}

Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å

Cell parameters from 6634 reflections

$\theta = 4.7–69.2°$

$\mu = 0.64$ mm^{-1}

$T = 173$ K

Needle, colourless

0.22 × 0.21 × 0.10 mm

Data collection

Bruker Kappa DUO APEXII diffractometer

Radiation source: fine-focus sealed tube

graphite

Detector resolution: n/a pixels mm^{-1}

0.5° φ scans and ω scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)

$T_{min} = 0.682$, $T_{max} = 0.753$

6634 measured reflections

1552 independent reflections

1479 reflections with $I > 2\sigma(I)$

$R_{int} = 0.036$

$\theta_{max} = 69.2°$, $\theta_{min} = 4.7°$

$h = -6 \rightarrow 6$

$k = -11 \rightarrow 12$

$l = -15 \rightarrow 30$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.076$

$S = 1.05$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 0.1838P]$

where $P = (F_o^2 + 2F_c^2)/3$

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

1552 reflections	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
195 parameters	$\Delta\rho_{min}$ = −0.12 e Å^{-3}
1 restraint	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0039 (5)

Special details

Experimental. Half sphere of data collected using *SAINT* strategy (Bruker, 2006). Crystal to detector distance = 50 mm; combination of φ and ω scans of 0.5°, 30 s per °, 2 iterations.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.4184 (2)	0.79986 (13)	0.19408 (5)	0.0306 (3)
N1	0.5780 (3)	1.06125 (15)	0.15598 (6)	0.0272 (3)
H1N	0.702 (3)	1.0028 (18)	0.1694 (7)	0.033 (6)*
N2	0.0533 (3)	0.90911 (15)	0.19216 (6)	0.0285 (4)
C1	0.6813 (4)	1.15005 (18)	0.11756 (7)	0.0324 (4)
H1A	0.8214	1.1991	0.1329	0.039*
H1B	0.5533	1.2157	0.1080	0.039*
C2	0.7714 (3)	1.08180 (18)	0.06969 (7)	0.0269 (4)
C3	0.9535 (4)	1.13952 (18)	0.03901 (7)	0.0322 (4)
H3	1.0184	1.2238	0.0480	0.039*
C4	1.0408 (4)	1.0762 (2)	−0.00419 (7)	0.0363 (5)
H4	1.1656	1.1162	−0.0245	0.044*
C5	0.9444 (4)	0.9538 (2)	−0.01748 (7)	0.0382 (5)
H5	1.0025	0.9096	−0.0471	0.046*
C6	0.7634 (4)	0.89605 (19)	0.01249 (7)	0.0356 (5)
H6	0.6978	0.8123	0.0031	0.043*
C7	0.6755 (4)	0.95876 (18)	0.05633 (7)	0.0285 (4)
C8	0.4854 (4)	0.89025 (19)	0.08944 (7)	0.0350 (5)
H8A	0.5606	0.8097	0.1043	0.042*
H8B	0.3448	0.8621	0.0678	0.042*
C9	0.3869 (3)	0.97793 (18)	0.13282 (7)	0.0272 (4)
H9	0.2567	1.0374	0.1183	0.033*
C10	0.2691 (3)	0.89535 (18)	0.17416 (7)	0.0260 (4)
C11	0.2612 (3)	0.72424 (17)	0.22836 (7)	0.0279 (4)

H11A	0.3444	0.7089	0.2615	0.033*
H11B	0.2159	0.6378	0.2132	0.033*
C12	0.0312 (3)	0.81315 (17)	0.23493 (7)	0.0258 (4)
H12	−0.1226	0.7592	0.2308	0.031*
C13	0.0251 (3)	0.88531 (17)	0.28547 (6)	0.0247 (4)
C14	−0.1619 (3)	0.86228 (18)	0.32043 (7)	0.0288 (4)
H14	−0.2902	0.8014	0.3124	0.035*
C15	−0.1639 (4)	0.92738 (19)	0.36719 (7)	0.0327 (4)
H15	−0.2932	0.9106	0.3909	0.039*
C16	0.0209 (4)	1.01606 (19)	0.37932 (7)	0.0329 (4)
H16	0.0188	1.0608	0.4112	0.040*
C17	0.2105 (4)	1.03964 (19)	0.34458 (7)	0.0330 (4)
H17	0.3394	1.0999	0.3528	0.040*
C18	0.2114 (3)	0.97527 (18)	0.29802 (7)	0.0290 (4)
H18	0.3403	0.9925	0.2743	0.035*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0268 (6)	0.0313 (6)	0.0336 (7)	0.0056 (6)	0.0025 (5)	0.0064 (5)
N1	0.0279 (7)	0.0272 (7)	0.0267 (7)	0.0012 (7)	0.0002 (6)	−0.0021 (6)
N2	0.0249 (7)	0.0349 (8)	0.0257 (7)	0.0035 (7)	−0.0025 (6)	0.0027 (7)
C1	0.0363 (11)	0.0260 (8)	0.0349 (10)	−0.0015 (8)	0.0026 (8)	−0.0034 (8)
C2	0.0281 (9)	0.0266 (8)	0.0260 (8)	0.0031 (8)	−0.0035 (7)	0.0029 (7)
C3	0.0335 (10)	0.0292 (9)	0.0338 (10)	−0.0020 (8)	−0.0014 (8)	0.0045 (8)
C4	0.0385 (10)	0.0396 (10)	0.0309 (9)	0.0013 (10)	0.0044 (8)	0.0087 (8)
C5	0.0493 (12)	0.0387 (10)	0.0267 (9)	0.0046 (10)	0.0070 (9)	0.0009 (8)
C6	0.0488 (12)	0.0302 (9)	0.0279 (9)	−0.0025 (9)	0.0006 (8)	−0.0024 (7)
C7	0.0326 (9)	0.0291 (9)	0.0237 (8)	0.0016 (8)	−0.0018 (7)	0.0029 (7)
C8	0.0437 (12)	0.0335 (9)	0.0277 (9)	−0.0099 (9)	0.0030 (8)	−0.0035 (8)
C9	0.0253 (9)	0.0310 (9)	0.0252 (9)	0.0017 (8)	−0.0027 (7)	0.0016 (7)
C10	0.0260 (9)	0.0272 (8)	0.0247 (8)	0.0024 (8)	−0.0050 (7)	−0.0001 (7)
C11	0.0297 (9)	0.0262 (8)	0.0278 (9)	0.0022 (8)	0.0005 (7)	0.0006 (7)
C12	0.0227 (8)	0.0284 (8)	0.0262 (8)	0.0007 (7)	−0.0006 (7)	−0.0002 (8)
C13	0.0232 (8)	0.0245 (8)	0.0264 (8)	0.0045 (7)	−0.0024 (7)	0.0029 (7)
C14	0.0254 (9)	0.0281 (9)	0.0329 (10)	−0.0005 (8)	0.0017 (8)	0.0027 (7)
C15	0.0311 (10)	0.0358 (10)	0.0313 (9)	0.0013 (9)	0.0059 (8)	0.0021 (8)
C16	0.0360 (11)	0.0361 (9)	0.0267 (8)	0.0044 (9)	−0.0025 (8)	−0.0049 (8)
C17	0.0320 (9)	0.0301 (9)	0.0368 (10)	−0.0044 (8)	−0.0050 (8)	−0.0035 (8)
C18	0.0244 (9)	0.0310 (9)	0.0316 (9)	−0.0010 (8)	0.0011 (7)	0.0030 (8)

Geometric parameters (Å, °)

O1—C10	1.362 (2)	C8—C9	1.537 (2)
O1—C11	1.454 (2)	C8—H8A	0.9900
N1—C1	1.460 (2)	C8—H8B	0.9900
N1—C9	1.464 (2)	C9—C10	1.508 (2)
N1—H1N	0.960 (10)	C9—H9	1.0000
N2—C10	1.265 (2)	C11—C12	1.542 (3)

N2—C12	1.487 (2)	C11—H11A	0.9900
C1—C2	1.513 (2)	C11—H11B	0.9900
C1—H1A	0.9900	C12—C13	1.513 (2)
C1—H1B	0.9900	C12—H12	1.0000
C2—C7	1.391 (3)	C13—C14	1.384 (2)
C2—C3	1.398 (3)	C13—C18	1.395 (3)
C3—C4	1.384 (3)	C14—C15	1.391 (3)
C3—H3	0.9500	C14—H14	0.9500
C4—C5	1.386 (3)	C15—C16	1.378 (3)
C4—H4	0.9500	C15—H15	0.9500
C5—C6	1.383 (3)	C16—C17	1.391 (3)
C5—H5	0.9500	C16—H16	0.9500
C6—C7	1.396 (3)	C17—C18	1.383 (3)
C6—H6	0.9500	C17—H17	0.9500
C7—C8	1.512 (3)	C18—H18	0.9500

C10—O1—C11	105.26 (13)	C10—C9—C8	111.06 (15)
C1—N1—C9	109.66 (14)	N1—C9—H9	108.0
C1—N1—H1N	111.3 (13)	C10—C9—H9	108.0
C9—N1—H1N	106.9 (13)	C8—C9—H9	108.0
C10—N2—C12	106.47 (15)	N2—C10—O1	118.72 (17)
N1—C1—C2	114.56 (14)	N2—C10—C9	126.58 (17)
N1—C1—H1A	108.6	O1—C10—C9	114.67 (15)
C2—C1—H1A	108.6	O1—C11—C12	103.52 (14)
N1—C1—H1B	108.6	O1—C11—H11A	111.1
C2—C1—H1B	108.6	C12—C11—H11A	111.1
H1A—C1—H1B	107.6	O1—C11—H11B	111.1
C7—C2—C3	119.32 (17)	C12—C11—H11B	111.1
C7—C2—C1	119.75 (16)	H11A—C11—H11B	109.0
C3—C2—C1	120.92 (17)	N2—C12—C13	110.39 (13)
C4—C3—C2	121.21 (18)	N2—C12—C11	103.33 (14)
C4—C3—H3	119.4	C13—C12—C11	113.32 (14)
C2—C3—H3	119.4	N2—C12—H12	109.9
C3—C4—C5	119.33 (19)	C13—C12—H12	109.9
C3—C4—H4	120.3	C11—C12—H12	109.9
C5—C4—H4	120.3	C14—C13—C18	118.68 (17)
C6—C5—C4	119.93 (19)	C14—C13—C12	121.02 (16)
C6—C5—H5	120.0	C18—C13—C12	120.29 (15)
C4—C5—H5	120.0	C13—C14—C15	120.66 (17)
C5—C6—C7	121.13 (19)	C13—C14—H14	119.7
C5—C6—H6	119.4	C15—C14—H14	119.7
C7—C6—H6	119.4	C16—C15—C14	120.32 (18)
C2—C7—C6	119.08 (18)	C16—C15—H15	119.8
C2—C7—C8	121.13 (16)	C14—C15—H15	119.8
C6—C7—C8	119.76 (17)	C15—C16—C17	119.57 (17)
C7—C8—C9	113.36 (15)	C15—C16—H16	120.2
C7—C8—H8A	108.9	C17—C16—H16	120.2
C9—C8—H8A	108.9	C18—C17—C16	119.98 (18)
C7—C8—H8B	108.9	C18—C17—H17	120.0
C9—C8—H8B	108.9	C16—C17—H17	120.0

H8A—C8—H8B	107.7	C17—C18—C13	120.78 (17)
N1—C9—C10	108.50 (14)	C17—C18—H18	119.6
N1—C9—C8	113.21 (15)	C13—C18—H18	119.6

C9—N1—C1—C2	−52.7 (2)	C11—O1—C10—C9	−173.78 (14)
N1—C1—C2—C7	23.6 (3)	N1—C9—C10—N2	108.6 (2)
N1—C1—C2—C3	−155.27 (17)	C8—C9—C10—N2	−126.4 (2)
C7—C2—C3—C4	−0.4 (3)	N1—C9—C10—O1	−69.47 (19)
C1—C2—C3—C4	178.49 (18)	C8—C9—C10—O1	55.6 (2)
C2—C3—C4—C5	0.5 (3)	C10—O1—C11—C12	−14.48 (17)
C3—C4—C5—C6	−0.3 (3)	C10—N2—C12—C13	109.57 (16)
C4—C5—C6—C7	−0.2 (3)	C10—N2—C12—C11	−11.91 (18)
C3—C2—C7—C6	−0.1 (3)	O1—C11—C12—N2	15.96 (17)
C1—C2—C7—C6	−178.97 (17)	O1—C11—C12—C13	−103.52 (16)
C3—C2—C7—C8	177.66 (17)	N2—C12—C13—C14	127.42 (17)
C1—C2—C7—C8	−1.2 (3)	C11—C12—C13—C14	−117.22 (18)
C5—C6—C7—C2	0.4 (3)	N2—C12—C13—C18	−53.6 (2)
C5—C6—C7—C8	−177.41 (18)	C11—C12—C13—C18	61.8 (2)
C2—C7—C8—C9	8.5 (3)	C18—C13—C14—C15	−0.1 (3)
C6—C7—C8—C9	−173.72 (17)	C12—C13—C14—C15	178.90 (16)
C1—N1—C9—C10	−175.40 (15)	C13—C14—C15—C16	0.1 (3)
C1—N1—C9—C8	60.83 (19)	C14—C15—C16—C17	−0.3 (3)
C7—C8—C9—N1	−38.5 (2)	C15—C16—C17—C18	0.7 (3)
C7—C8—C9—C10	−160.89 (15)	C16—C17—C18—C13	−0.7 (3)
C12—N2—C10—O1	3.0 (2)	C14—C13—C18—C17	0.4 (3)
C12—N2—C10—C9	−174.98 (16)	C12—C13—C18—C17	−178.58 (17)
C11—O1—C10—N2	8.0 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···N2^{i}	0.96 (1)	2.20 (1)	3.139 (2)	165 (2)

Symmetry codes: (i) *x*+1, *y*, *z*.

Fig. 1

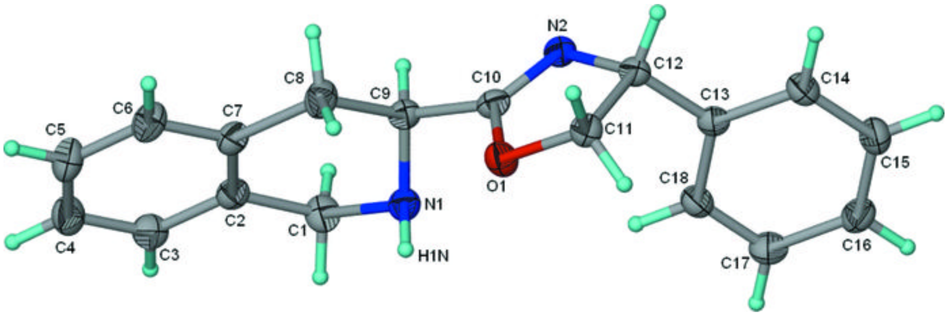
C8
C9
C10
N2
C12
C13
C14
C6
C7
C5
C11
C15
O1
C18
N1
C2
C1
C4
C3
H1N
C16
C17

Fig. 2