

# 1',3',3'-Trimethyl-2,3-diphenyl-2,3-dihydroisoxazole-5(4H)-spiro-2'-indoline

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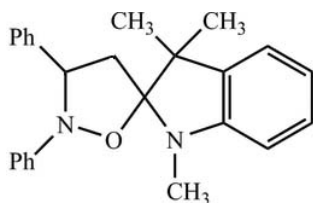
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Key indicators: single-crystal X-ray study;  $T = 180$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.042;  $wR$  factor = 0.105; data-to-parameter ratio = 7.9.

Two diastereoisomers of the title compound,  $\text{C}_{25}\text{H}_{26}\text{N}_2\text{O}$ , have been prepared by cycloaddition between 1,3,3-trimethyl-2-methyleneindoline and *C*-phenyl-*N*-phenylnitron. The stereochemistry of the major diastereoisomer, *viz.* *S,R/R,S*, is confirmed by the X-ray analysis. The oxazole and the pyrrole rings have envelope conformations. The packing is stabilized by weak  $\text{C}-\text{H}\cdots\pi$  interactions involving the phenyl ring attached to the N atom of the oxazole and the phenyl ring of the indole fragment.

## Related literature

For general background, see: Alonso-Perarnau *et al.* (1997); Cacciarini *et al.* (2000); Pariera *et al.* (1993). For related studies, see: Daran *et al.* (2006); Fihl *et al.* (1995, 2004); Roussel *et al.* (2000, 2003). For the synthetic procedure, see: Brüning *et al.* (1973). For puckering parameters, see: Cremer & Pople (1975).



## Experimental

### Crystal data

$\text{C}_{25}\text{H}_{26}\text{N}_2\text{O}$   
 $M_r = 370.48$   
Orthorhombic,  $Pna2_1$   
 $a = 18.0393$  (18) Å

$b = 8.9854$  (7) Å  
 $c = 12.3947$  (9) Å  
 $V = 2009.1$  (3) Å<sup>3</sup>  
 $Z = 4$

Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm<sup>-1</sup>

$T = 180$  (2) K  
 $0.48 \times 0.36 \times 0.28$  mm

### Data collection

Stoe IPDS diffractometer  
Absorption correction: none  
19030 measured reflections

2021 independent reflections  
1581 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.059$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.105$   
 $S = 1.15$   
2021 reflections  
256 parameters

1 restraint  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.26$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.15$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$Cg1$  is the centroid of the C21–C26 ring and  $Cg2$  is the centroid of the C3–C8 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C7-H7\cdots Cg1^i$	0.95	2.89	3.735 (3)	149
$C23-H23\cdots Cg2^{ii}$	0.95	2.95	3.803 (4)	150

Symmetry codes: (i)  $x, y + 1, z$ ; (ii)  $-x + 1, -y + 2, z - \frac{1}{2}$ .

Data collection: *IPDS* (Stoe & Cie, 2000); cell refinement: *IPDS*; data reduction: *X-RED* (Stoe & Cie, 1996); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP III* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2228).

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**supplementary materials**

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## 1',3',3'-Trimethyl-2,3-diphenyl-2,3-dihydroisoxazole-5(4*H*)-spiro-2'-indoline

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### Comment

Heterocyclic spirocompounds are of interest in synthetic organic chemistry (Pariera *et al.*, 1993; Alonso-Perarnau *et al.*, 1997; Cacciarini *et al.*, 2000). The cycloaddition between dipolarophiles bearing an exocyclic carbon-carbon double bond and appropriate 1,3-dipoles is one of the best methods for the synthesis of bicyclic spirocompounds.

As part of our research on bicyclic spirocompounds (Fihi *et al.*, 1995; Roussel *et al.*, 2000, 2003; Daran *et al.*, 2006), we reported that methylene lactones react with 1,3-dipoles with high selectivity. In a previous article (Fihi *et al.*, 2004) we reported that 1,3-dipolar cycloaddition of aryl nitriloxides and *N*-Phenylarylnitrilimines to 5-chloro-2-methylene-1,3,3-trimethylindoline is regiospecific. Arylnitriloxides reactions lead to spiroheterocyclic compounds, whereas *N*-phenylarylnitrilimines reactions afforded evolutive products.

We report here, the cycloaddition of *C*-phenyl-*N*-phenylnitrone (2) to 2-methylene-1,3,3-trimethylindoline (1). The reaction produced a mixture of diastereoisomers (Fig. 2). The ratio (77 / 23%) of which was evaluated by <sup>1</sup>H NMR (performed on the crude reaction mixture). To confirm unambiguously the structure assignment of (3) and (3'), and to establish the stereochemistry of each spiroheterocycle, an X-ray structural analysis was carried out on the major spirocompound, because the <sup>1</sup>H and <sup>13</sup>C NMR studies did not provide unambiguous information.

The stereochemistry of the major diastereoisomer, *S,R/R,S*, is confirmed by the X-ray analysis (Fig. 1). The oxazole and the pyrrole rings have an envelope conformation with puckering parameters  $Q(2) = 0.399$  (3)°,  $\varphi(2) = 218.9$  (5)° and  $Q(2) = 0.274$  (3)°,  $\varphi(2) = 218.9$  (7)° (Cremer & Pople, 1975). The packing is stabilized by weak C—H... $\pi$  interactions involving the phenyl attached to the nitrogen of the oxazole and the phenyl of the indole fragment (Table 1: Cg1 is the centroid of the C21—C26 ring and Cg2 is the centroid of the C3—C8 ring).

### Experimental

2-Methylene-1,3,3-trimethylindoline (1) is a commercial product. *C*-phenyl, *N*-diphenylnitrone (2) was synthesized according to the literature procedure (Brüning *et al.*, 1973). A solution of (2) (1 g, 6 mmol), (1) (1.12 g, 6 mmol) in ethylacetate (40 ml) was stirred at reflux for 24 h. The solvent was then evaporated under reduced pressure. The residue was crystallized from ethanol, leading to a mixture of diastereoisomers (3) and (3'). They were separated and purified by chromatography on silica gel (eluant: dichloromethane / hexane: 10 / 90). The spirocompounds were finally recrystallized from dichloromethane.

### Refinement

All H atoms were fixed geometrically and treated as riding on their parent atoms with C—H = 0.98 Å (methyl), 0.99 Å (methylene), 1.0 Å (methine) and 0.95 Å (aromatic) with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{aromatic, methylene and methine})$  or  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl})$ .

# supplementary materials

In the absence of significant anomalous scattering, the absolute structure could not be reliably determined and the Friedel pairs were merged and any references to the Flack parameter were removed.

## Figures

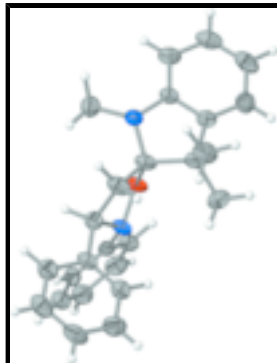


Fig. 1. Molecular view of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented as small spheres of arbitrary radii.



Fig. 2. The formation of the title compound.

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### Crystal data

$C_{25}H_{26}N_2O$   
 $M_r = 370.48$

Orthorhombic,  $Pna2_1$

Hall symbol: P 2c -2n

$a = 18.0393$  (18) Å

$b = 8.9854$  (7) Å

$c = 12.3947$  (9) Å

$V = 2009.1$  (3) Å<sup>3</sup>

$Z = 4$

$F_{000} = 792$

$D_x = 1.225$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 8000 reflections

$\theta = 1.7$ – $26.2^\circ$

$\mu = 0.08$  mm<sup>-1</sup>

$T = 180$  (2) K

Prism, colorless

$0.48 \times 0.36 \times 0.28$  mm

### Data collection

Stoe IPDS  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 180$ (2) K

$\varphi$  scans

Absorption correction: none

19030 measured reflections

2021 independent reflections

1581 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.059$

$\theta_{max} = 26.0^\circ$

$\theta_{min} = 2.5^\circ$

$h = -22 \rightarrow 22$

$k = -11 \rightarrow 11$

$l = -14 \rightarrow 14$

*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-atom parameters constrained
$wR(F^2) = 0.105$	$w = 1/[\sigma^2(F_o^2) + (0.061P)^2]$
$S = 1.15$	where $P = (F_o^2 + 2F_c^2)/3$
2021 reflections	$(\Delta/\sigma)_{\max} = 0.007$
256 parameters	$\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

*Special details*

**Experimental.** The data were collected on a Stoe Imaging Plate Diffraction System (*IPDS*). The crystal-to-detector distance was 70 mm. 167 frames (4 min per frame) were obtained with  $0 < \varphi < 250.5^\circ$  and with the crystals rotated through  $1.5^\circ$  in  $\varphi$ . Coverage of the unique set was over 97.4% complete to at least  $26.04^\circ$ . Crystal decay was monitored by measuring 200 reflections per frame.

**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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.43438 (17)	0.9280 (3)	0.0510 (3)	0.0360 (7)
C2	0.52004 (18)	0.9356 (3)	0.0701 (3)	0.0398 (7)
C3	0.53941 (16)	1.0776 (3)	0.0091 (3)	0.0346 (7)
C4	0.60643 (18)	1.1346 (4)	-0.0231 (3)	0.0470 (8)
H4	0.6508	1.0802	-0.0111	0.056*
C5	0.6086 (2)	1.2734 (4)	-0.0738 (3)	0.0523 (9)
H5	0.6547	1.3141	-0.0964	0.063*
C6	0.5439 (2)	1.3510 (4)	-0.0909 (3)	0.0482 (9)
H6	0.5460	1.4452	-0.1256	0.058*
C7	0.47568 (18)	1.2952 (3)	-0.0588 (3)	0.0412 (7)
H7	0.4312	1.3493	-0.0710	0.049*
C8	0.47495 (15)	1.1576 (3)	-0.0082 (3)	0.0327 (7)
C9	0.34450 (16)	0.7260 (3)	0.0744 (3)	0.0363 (7)
H9	0.2955	0.7656	0.0500	0.044*

## supplementary materials

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C10	0.38766 (19)	0.8462 (4)	0.1354 (3)	0.0458 (8)
H10A	0.3531	0.9161	0.1713	0.055*
H10B	0.4199	0.8006	0.1908	0.055*
C21	0.36716 (14)	0.6294 (3)	-0.1120 (2)	0.0294 (6)
C22	0.40480 (18)	0.6491 (3)	-0.2083 (3)	0.0367 (7)
H22	0.4453	0.7164	-0.2117	0.044*
C23	0.38362 (18)	0.5708 (3)	-0.2998 (3)	0.0429 (7)
H23	0.4091	0.5856	-0.3660	0.051*
C24	0.32514 (17)	0.4710 (4)	-0.2943 (3)	0.0438 (8)
H24	0.3110	0.4163	-0.3565	0.053*
C25	0.28787 (18)	0.4513 (4)	-0.1993 (3)	0.0414 (8)
H25	0.2482	0.3821	-0.1959	0.050*
C26	0.30746 (15)	0.5317 (3)	-0.1074 (3)	0.0352 (7)
H26	0.2803	0.5199	-0.0423	0.042*
C91	0.33383 (16)	0.5862 (3)	0.1405 (3)	0.0359 (7)
C92	0.39087 (17)	0.4871 (3)	0.1574 (3)	0.0418 (8)
H92	0.4380	0.5056	0.1259	0.050*
C93	0.3806 (2)	0.3610 (4)	0.2195 (3)	0.0511 (9)
H93	0.4205	0.2936	0.2302	0.061*
C94	0.3131 (2)	0.3330 (4)	0.2658 (3)	0.0537 (9)
H94	0.3064	0.2460	0.3082	0.064*
C95	0.2549 (2)	0.4299 (4)	0.2513 (3)	0.0532 (9)
H95	0.2083	0.4109	0.2841	0.064*
C96	0.26512 (18)	0.5563 (4)	0.1878 (3)	0.0462 (8)
H96	0.2249	0.6229	0.1767	0.055*
C111	0.33893 (17)	1.1202 (4)	0.0002 (4)	0.0526 (9)
H11A	0.3290	1.2248	0.0175	0.079*
H11B	0.3032	1.0567	0.0380	0.079*
H11C	0.3342	1.1051	-0.0778	0.079*
C211	0.5371 (2)	0.9600 (4)	0.1901 (3)	0.0531 (9)
H21A	0.5894	0.9871	0.1986	0.080*
H21B	0.5270	0.8682	0.2301	0.080*
H21C	0.5058	1.0403	0.2181	0.080*
C212	0.5604 (2)	0.7992 (4)	0.0285 (4)	0.0576 (11)
H21D	0.5484	0.7845	-0.0479	0.086*
H21E	0.5448	0.7117	0.0698	0.086*
H21F	0.6139	0.8134	0.0366	0.086*
N1	0.41386 (13)	1.0819 (2)	0.0341 (2)	0.0369 (6)
N2	0.39329 (13)	0.7023 (2)	-0.0180 (2)	0.0323 (6)
O1	0.41789 (11)	0.8501 (2)	-0.04964 (17)	0.0370 (5)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0486 (17)	0.0268 (14)	0.0327 (18)	-0.0061 (13)	0.0006 (14)	-0.0026 (13)
C2	0.0508 (18)	0.0289 (15)	0.040 (2)	0.0018 (13)	-0.0082 (15)	0.0004 (13)
C3	0.0405 (16)	0.0306 (15)	0.0327 (19)	-0.0027 (12)	0.0001 (13)	0.0003 (12)
C4	0.0412 (17)	0.0488 (19)	0.051 (2)	-0.0023 (14)	0.0057 (15)	-0.0044 (17)

C5	0.061 (2)	0.052 (2)	0.045 (2)	-0.0220 (17)	0.0183 (17)	-0.0051 (16)
C6	0.075 (2)	0.0369 (18)	0.033 (2)	-0.0124 (16)	0.0080 (16)	-0.0010 (14)
C7	0.0545 (18)	0.0294 (16)	0.040 (2)	-0.0016 (13)	-0.0020 (15)	0.0007 (14)
C8	0.0393 (15)	0.0278 (15)	0.0308 (17)	-0.0033 (11)	0.0016 (13)	-0.0016 (12)
C9	0.0392 (16)	0.0353 (15)	0.0344 (19)	-0.0024 (13)	0.0062 (13)	-0.0029 (12)
C10	0.0589 (19)	0.0389 (17)	0.040 (2)	-0.0125 (14)	0.0075 (16)	-0.0062 (15)
C21	0.0326 (14)	0.0234 (13)	0.0322 (18)	0.0021 (11)	-0.0030 (12)	-0.0014 (12)
C22	0.0439 (16)	0.0333 (15)	0.0328 (19)	-0.0024 (12)	0.0017 (13)	0.0000 (13)
C23	0.0537 (18)	0.0432 (17)	0.0318 (18)	0.0047 (14)	0.0002 (15)	-0.0054 (15)
C24	0.0491 (18)	0.0433 (17)	0.039 (2)	0.0008 (13)	-0.0094 (16)	-0.0094 (15)
C25	0.0419 (16)	0.0399 (16)	0.042 (2)	-0.0044 (13)	-0.0055 (15)	-0.0063 (15)
C26	0.0323 (14)	0.0364 (15)	0.0369 (18)	-0.0013 (12)	-0.0045 (13)	0.0005 (14)
C91	0.0445 (16)	0.0323 (15)	0.0307 (17)	-0.0099 (13)	0.0010 (14)	-0.0021 (13)
C92	0.0427 (16)	0.0409 (17)	0.042 (2)	-0.0040 (13)	-0.0009 (14)	-0.0015 (15)
C93	0.061 (2)	0.0437 (19)	0.048 (2)	-0.0014 (15)	-0.0055 (17)	0.0017 (16)
C94	0.066 (2)	0.0429 (19)	0.052 (2)	-0.0193 (17)	-0.0025 (18)	0.0060 (17)
C95	0.054 (2)	0.058 (2)	0.047 (2)	-0.0182 (18)	0.0122 (17)	0.0016 (18)
C96	0.0472 (17)	0.0453 (18)	0.046 (2)	-0.0044 (14)	0.0023 (16)	-0.0026 (16)
C111	0.0396 (17)	0.0475 (18)	0.071 (3)	0.0017 (14)	-0.0049 (18)	-0.0098 (18)
C211	0.066 (2)	0.0489 (19)	0.045 (2)	-0.0085 (16)	-0.0146 (18)	0.0061 (17)
C212	0.055 (2)	0.0375 (18)	0.081 (3)	0.0098 (16)	-0.0048 (19)	-0.0012 (18)
N1	0.0374 (13)	0.0269 (12)	0.0464 (17)	-0.0029 (11)	0.0025 (11)	-0.0012 (12)
N2	0.0410 (13)	0.0248 (12)	0.0311 (15)	-0.0095 (10)	0.0016 (10)	-0.0016 (11)
O1	0.0570 (13)	0.0239 (10)	0.0302 (12)	-0.0113 (8)	0.0029 (10)	-0.0018 (9)

*Geometric parameters (Å, °)*

C1—N1	1.446 (4)	C23—C24	1.386 (5)
C1—O1	1.461 (4)	C23—H23	0.9500
C1—C10	1.531 (5)	C24—C25	1.367 (5)
C1—C2	1.565 (4)	C24—H24	0.9500
C2—C212	1.516 (5)	C25—C26	1.395 (4)
C2—C3	1.523 (4)	C25—H25	0.9500
C2—C211	1.534 (5)	C26—H26	0.9500
C3—C4	1.372 (4)	C91—C92	1.377 (4)
C3—C8	1.384 (4)	C91—C96	1.397 (4)
C4—C5	1.397 (5)	C92—C93	1.382 (5)
C4—H4	0.9500	C92—H92	0.9500
C5—C6	1.376 (5)	C93—C94	1.369 (6)
C5—H5	0.9500	C93—H93	0.9500
C6—C7	1.388 (5)	C94—C95	1.375 (6)
C6—H6	0.9500	C94—H94	0.9500
C7—C8	1.387 (4)	C95—C96	1.393 (5)
C7—H7	0.9500	C95—H95	0.9500
C8—N1	1.397 (4)	C96—H96	0.9500
C9—N2	1.460 (4)	C111—N1	1.457 (4)
C9—C91	1.512 (4)	C111—H11A	0.9800
C9—C10	1.531 (4)	C111—H11B	0.9800
C9—H9	1.0000	C111—H11C	0.9800

## supplementary materials

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C10—H10A	0.9900	C211—H21A	0.9800
C10—H10B	0.9900	C211—H21B	0.9800
C21—C22	1.384 (4)	C211—H21C	0.9800
C21—C26	1.390 (4)	C212—H21D	0.9800
C21—N2	1.417 (4)	C212—H21E	0.9800
C22—C23	1.388 (5)	C212—H21F	0.9800
C22—H22	0.9500	N2—O1	1.454 (3)
N1—C1—O1	106.4 (2)	C25—C24—H24	120.0
N1—C1—C10	114.6 (3)	C23—C24—H24	120.0
O1—C1—C10	104.0 (2)	C24—C25—C26	120.8 (3)
N1—C1—C2	103.5 (2)	C24—C25—H25	119.6
O1—C1—C2	110.6 (2)	C26—C25—H25	119.6
C10—C1—C2	117.4 (3)	C21—C26—C25	119.3 (3)
C212—C2—C3	113.4 (3)	C21—C26—H26	120.3
C212—C2—C211	110.5 (3)	C25—C26—H26	120.3
C3—C2—C211	108.4 (3)	C92—C91—C96	118.4 (3)
C212—C2—C1	112.8 (3)	C92—C91—C9	121.6 (3)
C3—C2—C1	100.8 (2)	C96—C91—C9	120.0 (3)
C211—C2—C1	110.6 (3)	C91—C92—C93	120.9 (3)
C4—C3—C8	120.1 (3)	C91—C92—H92	119.5
C4—C3—C2	131.2 (3)	C93—C92—H92	119.5
C8—C3—C2	108.6 (3)	C94—C93—C92	120.2 (3)
C3—C4—C5	119.3 (3)	C94—C93—H93	119.9
C3—C4—H4	120.4	C92—C93—H93	119.9
C5—C4—H4	120.4	C93—C94—C95	120.5 (3)
C6—C5—C4	119.9 (3)	C93—C94—H94	119.8
C6—C5—H5	120.1	C95—C94—H94	119.8
C4—C5—H5	120.1	C94—C95—C96	119.3 (3)
C5—C6—C7	121.7 (3)	C94—C95—H95	120.3
C5—C6—H6	119.2	C96—C95—H95	120.3
C7—C6—H6	119.2	C95—C96—C91	120.7 (3)
C8—C7—C6	117.4 (3)	C95—C96—H96	119.7
C8—C7—H7	121.3	C91—C96—H96	119.7
C6—C7—H7	121.3	N1—C111—H11A	109.5
C3—C8—C7	121.7 (3)	N1—C111—H11B	109.5
C3—C8—N1	110.6 (3)	H11A—C111—H11B	109.5
C7—C8—N1	127.7 (3)	N1—C111—H11C	109.5
N2—C9—C91	112.4 (2)	H11A—C111—H11C	109.5
N2—C9—C10	100.6 (2)	H11B—C111—H11C	109.5
C91—C9—C10	112.6 (3)	C2—C211—H21A	109.5
N2—C9—H9	110.3	C2—C211—H21B	109.5
C91—C9—H9	110.3	H21A—C211—H21B	109.5
C10—C9—H9	110.3	C2—C211—H21C	109.5
C1—C10—C9	106.4 (3)	H21A—C211—H21C	109.5
C1—C10—H10A	110.5	H21B—C211—H21C	109.5
C9—C10—H10A	110.5	C2—C212—H21D	109.5
C1—C10—H10B	110.5	C2—C212—H21E	109.5
C9—C10—H10B	110.5	H21D—C212—H21E	109.5
H10A—C10—H10B	108.6	C2—C212—H21F	109.5



C22—C21—C26	119.7 (3)	H21D—C212—H21F	109.5
C22—C21—N2	119.1 (2)	H21E—C212—H21F	109.5
C26—C21—N2	121.0 (3)	C8—N1—C1	108.5 (2)
C21—C22—C23	120.3 (3)	C8—N1—C111	120.6 (3)
C21—C22—H22	119.8	C1—N1—C111	120.4 (2)
C23—C22—H22	119.8	C21—N2—O1	107.6 (2)
C24—C23—C22	119.8 (3)	C21—N2—C9	120.8 (2)
C24—C23—H23	120.1	O1—N2—C9	105.2 (2)
C22—C23—H23	120.1	N2—O1—C1	105.6 (2)
C25—C24—C23	120.0 (3)		

*Hydrogen-bond geometry* ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7—H7 $\cdots$ Cg1 <sup>i</sup>	0.95	2.89	3.735 (3)	149
C23—H23 $\cdots$ Cg2 <sup>ii</sup>	0.95	2.95	3.803 (4)	150

Symmetry codes: (i)  $x, y+1, z$ ; (ii)  $-x+1, -y+2, z-1/2$ .

Fig. 1

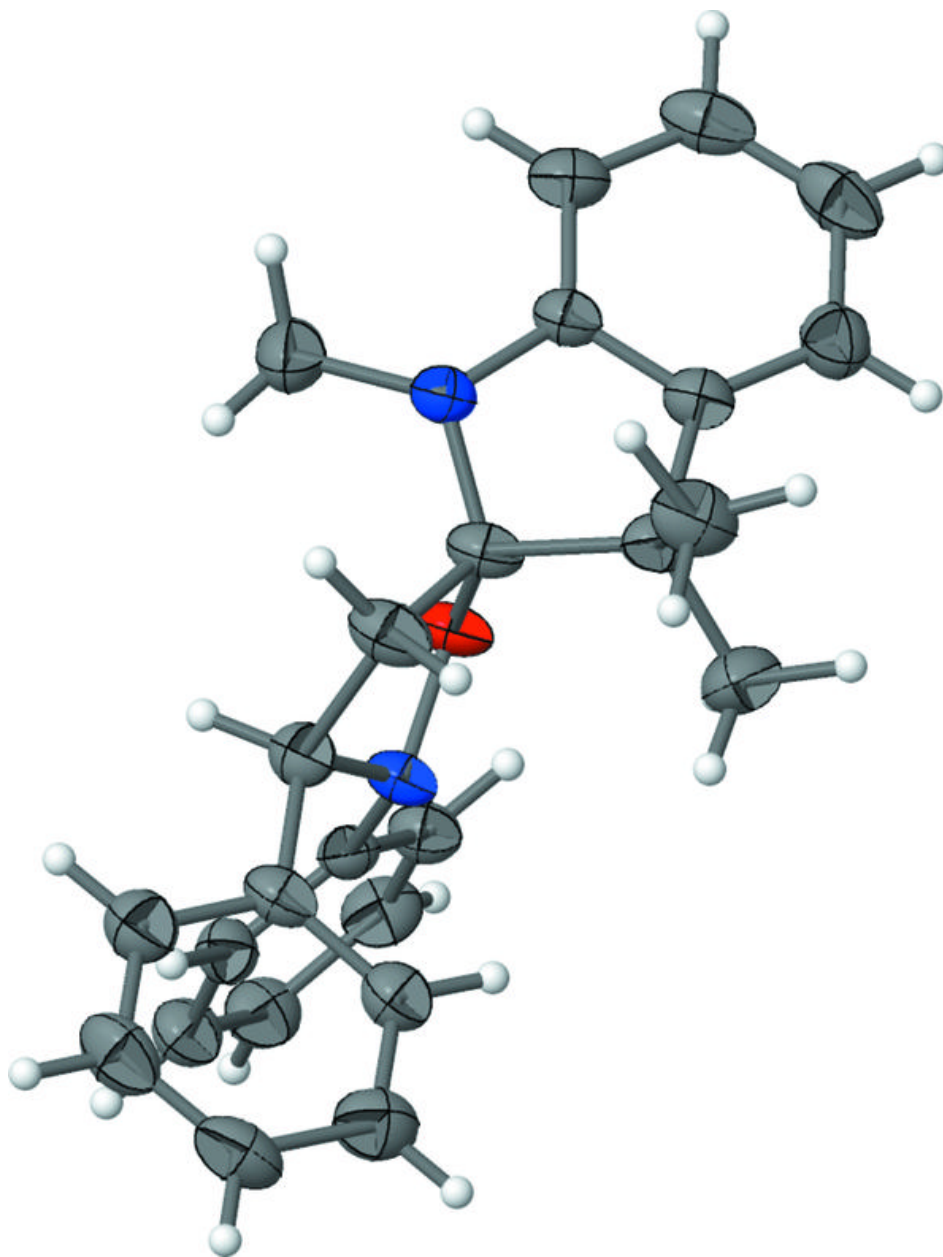


Fig. 2

