Acta Crystallographica Section E
**Structure Reports**
**Online**

ISSN 1600-5368

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

**Naoual Laghrib,[a] Jean-Claude Daran,[b] Rachid Fihi,[a] Lhou Majidi[a] and Mohamed Azrour[c]***

[a]Laboratoire des Substances Naturelles & Synthèse et Dynamique Moléculaire, Faculté des Sciences et Techniques, BP 509, Errachidia, Morocco, [b]Laboratoire de Chimie de Coordination, UPR–CNRS 8241, 205 route de Narbonne, 31077 Toulouse Cedex, France, and [c]Laboratoire de Physico-Chimie des Matèriaux, Faculté des Sciences et Techniques, BP 509, Errachidia, Morocco
Correspondence e-mail: mohamedazrour@yahoo.fr

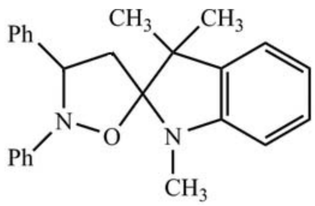
Received 30 December 2008; accepted 15 January 2009

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

Two diastereoisomers of the title compound, $C_{25}H_{26}N_2O$, have been prepared by cycloaddition between 1,3,3-trimethyl-2-methyleneindoline and *C*-phenyl-*N*-phenylnitrone. The stereochemistry of the major diastereoisomer, *viz. S,R*/*R,S*, is confirmed by the X-ray analysis. The oxazole and the pyrole rings have envelope conformations. The packing is stabilized by weak C—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); Fihi *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*

$C_{25}H_{26}N_2O$
$M_r$ = 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$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 180 (2) K
0.48 × 0.36 × 0.28 mm

*Data collection*

Stoe IPDS diffractometer
Absorption correction: none
19030 measured reflections
2021 independent reflections
1581 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 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
$\Delta\rho_{\rm max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{\rm min}$ = −0.15 e Å$^{-3}$

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

*Cg*1is the centroid of the C21–C26 ring and *Cg*2 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: *ORTEPIII* (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).

## References

Alonso-Perarnau, D., De March, P., El Arrad, M., Figueredo, M., Font, J. & Parella, T. (1997). *Tetrahedron*, **53**, 14763–14772.
Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Brüning, I., Grashey, R., Hauck, H., Huisgen, R. & Seidel, H. (1973). *Org. Synth. Coll.* **V**, pp. 1124–1129.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Cacciarini, M., Cordero, M., Faggi, C. & Goti, A. (2000). *Molecules*, **5**, 637–647.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Daran, J.-C., Fihi, R., Roussel, C., Laghrib, N., Azrour, M., Ciamala, K. & Vebreld, J. (2006). *Acta Cryst.* E**62**, o329–o331.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Fihi, R., Ciamala, K., Vebrel, J. & Rodier, N. (1995). *Bull. Soc. Chim. Belg.* **104**, 1, 55–62.
Fihi, R., Majidi, L. & Zair, T. (2004). *J. Mar. Chim. Heterocycl.* **3**, 52–56.
Pariera, S. M., Savage, J. P., Simpson, G. W., Greenword, R. J. & Mackay, M. F. (1993). *Tetrahedron*, **6**, 1401–1412.
Roussel, C., Fihi, R., Ciamala, K., Audebert, P. & Vebrel, J. (2000). *New J. Chem.* **24**, 471–476.
Roussel, C., Fihi, R., Ciamala, K., Vebrel, J., Zair, T. & Riche, C. (2003). *Org. Biomol. Chem.* **1**, 2689–2698.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Stoe & Cie (1996). *X-RED*. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (2000). *IPDS Manual*. Stoe & Cie, Darmstadt, Germany.

# supporting information

*Acta Cryst.* (2009). E**65**, o374 [doi:10.1107/S1600536809002062]

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

**Naoual Laghrib, Jean-Claude Daran, Rachid Fihi, Lhou Majidi and Mohamed Azrour**

### S1. 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 appropriate1,3-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 arylnitriloxydes and *N*-Phenylarylnitrilimines to 5-chloro-2-methylene-1,3,3-trimethylindoline is regiospecific. Arylnitriloxydes reactions lead to spiroheterocyclic compounds, whereas *N*-phenylarylnitrilimines reactions afforded evolutives 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 $^1$HNMR (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 analyses was carried out on the major spiro-compound, because the $^1$H and $^{13}$CNMR studies did not provide unambiguous information.

The stereochemistry of the major diastereoisomer, *S,R*/*R,S*, is confirmed by the X-ray analyses (Fig. 1). The oxazole and the pyrole 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: *Cg*1is the centroid of the C21—C26 ring and *Cg*2 is the centroid of the C3—C8 ring).

### S2. 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 ethyl-acetate (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.

### S3. 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 $U_{iso}$(H) = 1.2$U_{eq}$(aromatic, methylene and methine) or $U_{iso}$(H) = 1.5$U_{eq}$(methyl).

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.

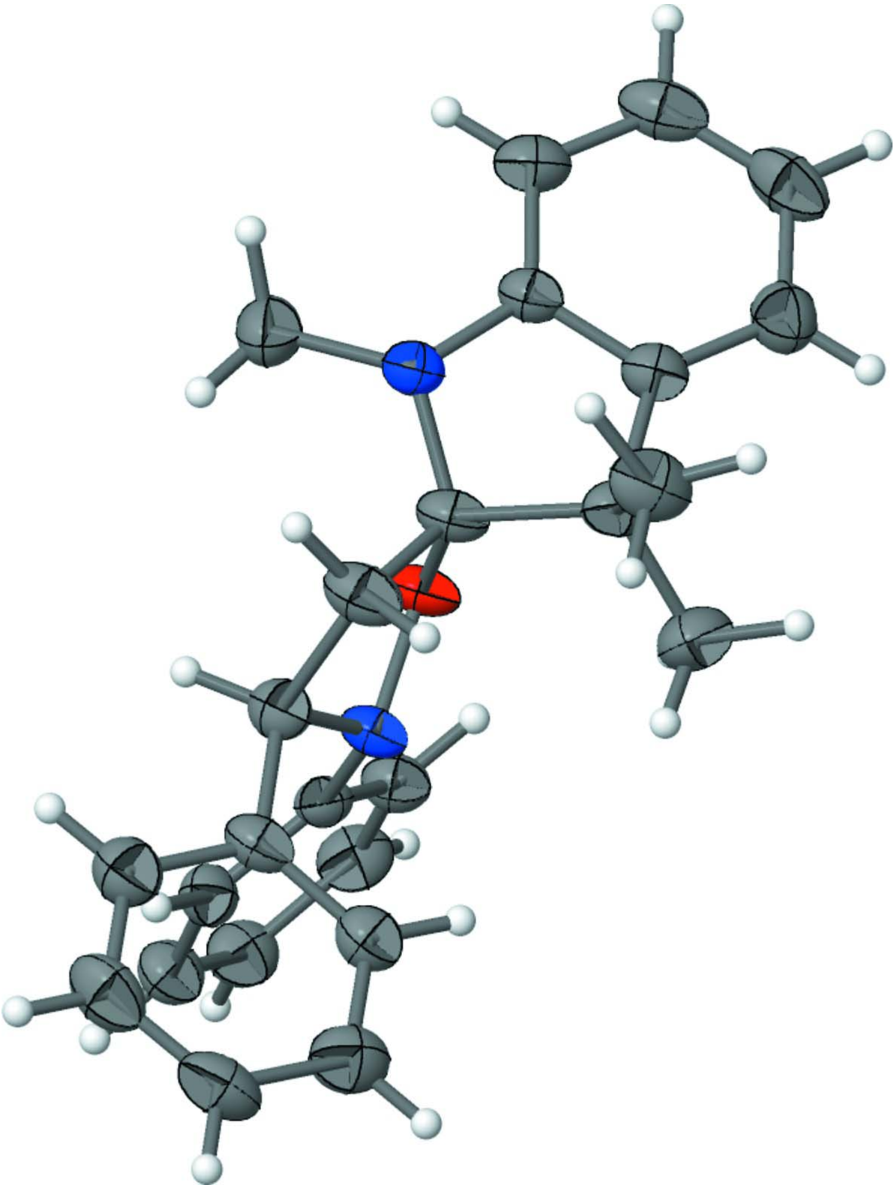


**Figure 1**

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

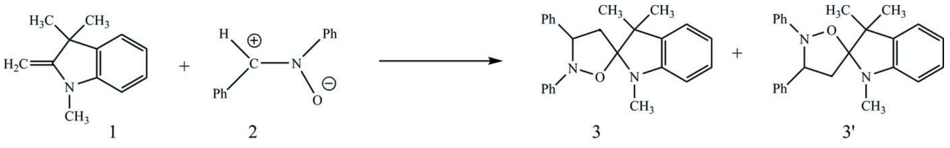


**Figure 2**

The formation of the title compound.

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

*Crystal data*

| | |
|---|---|
| $C_{25}H_{26}N_2O$ | $F(000) = 792$ |
| $M_r = 370.48$ | $D_x = 1.225$ Mg m$^{-3}$ |
| Orthorhombic, $Pna2_1$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: P 2c -2n | Cell parameters from 8000 reflections |
| $a = 18.0393$ (18) Å | $\theta = 1.7$–26.2° |
| $b = 8.9854$ (7) Å | $\mu = 0.08$ mm$^{-1}$ |
| $c = 12.3947$ (9) Å | $T = 180$ K |
| $V = 2009.1$ (3) Å$^3$ | Prism, colorless |
| $Z = 4$ | 0.48 × 0.36 × 0.28 mm |

*Data collection*

| | |
|---|---|
| Stoe IPDS diffractometer | 1581 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{\rm int} = 0.059$ |
| Graphite monochromator | $\theta_{\rm max} = 26.0°$, $\theta_{\rm min} = 2.5°$ |
| $\varphi$ scans | $h = -22\rightarrow22$ |
| 19030 measured reflections | $k = -11\rightarrow11$ |
| 2021 independent reflections | $l = -14\rightarrow14$ |

*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 |
| $wR(F^2) = 0.105$ | $w = 1/[\sigma^2(F_o^2) + (0.061P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.15$ | $(\Delta/\sigma)_{\rm max} = 0.007$ |
| 2021 reflections | $\Delta\rho_{\rm max} = 0.26$ e Å$^{-3}$ |
| 256 parameters | $\Delta\rho_{\rm min} = -0.15$ e Å$^{-3}$ |
| 1 restraint | |
| Primary atom site location: structure-invariant direct methods | |

*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 $0 < \varphi < 250.5°$ and with the crystals rotated through 1.5° in $\varphi$. Coverage of the unique set was over 97.4% complete to at least 26.04°. 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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{\rm iso}$*/$U_{\rm eq}$ |
|---|---|---|---|---|
| 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* |
| 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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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 |
| 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 (Å, º)*

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

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