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# *N*-Phenyl-2-(phenyliminomethyl)pyrrole-1-carboxamide

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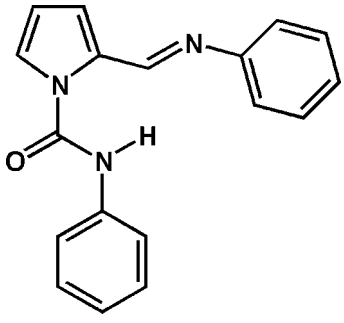
Key indicators: single-crystal X-ray study; $T$ = 183 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.041; $wR$ factor = 0.082; data-to-parameter ratio = 16.4.

The title compound, $C_{18}H_{15}N_3O$, was prepared from phenyl-(1*H*-pyrrol-2-ylmethylene)amine and phenyl isocyanate in the presence of catalytic amounts of $[Pd(PPh_3)_4]$. The conformation of the molecular structure is determined by an intramolecular hydrogen bond between the amide NH function and the imine N atom. The molecule is essentially planar. Only the peripheral phenyl substitutents are bent out of the plane.

## Related literature

For related literature, see: Mishriky *et al.* (1998).




## Experimental

### *Crystal data*

$C_{18}H_{15}N_3O$
$M_r$ = 289.33
Orthorhombic, *Pccn*
$a$ = 18.292 (2) Å
$b$ = 19.966 (2) Å
$c$ = 7.8479 (4) Å
$V$ = 2866.1 (4) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 183 (2) K
0.15 × 0.1 × 0.1 mm

### *Data collection*

Nonius KappaCCD diffractometer
Absorption correction: none
11352 measured reflections
3270 independent reflections
1391 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.082

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.082
$S$ = 0.74
3270 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H3N···N2 | 0.88 | 1.83 | 2.691 (2) | 166 |

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1990); software used to prepare material for publication: *SHELXL97* and *XP*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2518).

## References

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# supplementary materials

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# *N*-Phenyl-2-(phenyliminomethyl)pyrrole-1-carboxamide

**W. Imhof**

## Comment

Derivatives of the title compound have only been described once as intermediates in the synthesis of 1*H*-pyrrolo[1,2-*c*]imidazoles which were synthesized because of their molluscicidal activity (Mishriky *et al.*, 1998). To the best of our knowledge none of these derivatives have been structurally characterized.

The title compound is produced by the formal insertion of an isocyanate into the N—H bond of the pyrrol ring. The conformation of the amide and the imine substituent relative to each other is determined by an intramolecular N3—H3N···N2 hydrogen bond between the amide NH function and the imine nitrogen atom. The other bond lengths and angles are of expected values.

## Experimental

230 mg (1.35 mmol) Phenyl-(1*H*-pyrrol-2-ylmethylene)-amine and 240 mg (2.025 mmol) phenylisocyanate were refluxed in 20 ml of THF together with 65 mg (0.056 mmol) [$Pd(PPh_3)_4$] and 4 mg glacial acetic acid for 2 hrs. Evaporation of the solvent yielded an orange oil. Column chromatography on silica yielded 90 mg (23%) of the title compound using a mixture of pentane and ethyl acetate (5:1) as the eluent. With a ratio of 2:1 a compound which corresponding to it's mass spectrum is composed of one equivalent of imine and two equivalents of isocyanate was obtained (40 mg, 7%). Colorless crystals of the title compound were produced from a solution in a pentane/ethyl acetate mixture (10:1) at −20 °.

MS (EI) [m/*z* (%)]: 289 ($M^+$, 4), 197 ($C_{12}H_9N_2O^+$, 40), 169 ($C_{11}H_9N_2^+$, 100), 119 ($PhNCO^+$, 76), 91 ($C_7H_7^+$, 41), 77 ($C_6H_5^+$, 28), 64 ($C_5H_4^+$, 16), 51 ($C_4H_3^+$, 15), 39 ($C_3H_3^+$, 10; $^1$H-NMR ($CDCl_3$, 293 K) [p.p.m.]: 6.44–6.48 (m, 2 H, $CH_{pyrrole}$), 7.01–7.05 (m, 1H, $CH_{pyrrole}$), 7.18–7.77 (m, 8H, $CH_{ar}$), 8.21–8.23 (m, 2H, $CH_{ar}$), 8.38 (s, 1H, $CH_{imine}$), 14.74 (s, 1H, $CH_{amide}$); $^{13}$C-NMR ($CDCl_3$, 293 K) [p.p.m.]: 110.7, 120.7, 120.9, 124.1, 126.6, 128.1, 129.0, 129.6, 130.3, 131.4, 138.4, 148.2, 148.8, 151.3.

## Refinement

Hydrogen atoms were calculated in idealized positions and refined with distances of 0.88 Å (N3—H3N) and 0.95 Å (C—H). All hydrogen atoms were refined using a riding model with $U_{iso}$(H) = 1.5 times $U_{iso}$(C, N).

## Figures

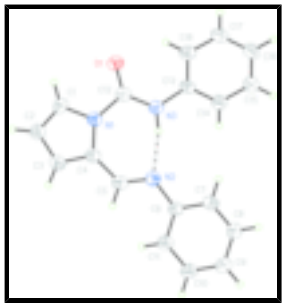


Fig. 1. The molecular structure of the title compoud showing the labelling scheme. Displacement ellipsoids are presented at the 40% probalitiy level. The dashed line indicates a hydrogen bond.

## *N*-Phenyl-2-(phenyliminomethyl)pyrrole-1-carboxamide

### *Crystal data*

| | |
|---|---|
| $C_{18}H_{15}N_3O$ | $F_{000}$ = 1216 |
| $M_r$ = 289.33 | $D_x$ = 1.341 Mg m$^{-3}$ |
| Orthorhombic, *Pccn* | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2ab 2ac | Cell parameters from 11352 reflections |
| *a* = 18.292 (2) Å | θ = 3.0–27.5º |
| *b* = 19.966 (2) Å | μ = 0.09 mm$^{-1}$ |
| *c* = 7.8479 (4) Å | *T* = 183 (2) K |
| *V* = 2866.1 (4) Å$^3$ | Cuboid, colorless |
| *Z* = 8 | 0.15 × 0.1 × 0.1 mm |

### *Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 1391 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int}$ = 0.082 |
| Monochromator: graphite | $\theta_{max}$ = 27.5º |
| *T* = 183(2) K | $\theta_{min}$ = 3.0º |
| φ and ω scans | *h* = −23→23 |
| Absorption correction: none | *k* = −25→25 |
| 11352 measured reflections | *l* = −8→10 |
| 3270 independent reflections | |

### *Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.041 | H-atom parameters constrained |
| $wR(F^2)$ = 0.082 | $w = 1/[\sigma^2(F_o^2) + (0.0346P)^2 + 0.003P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |

| | |
|---|---|
| $S$ = 0.74 | $(\Delta/\sigma)_{max}$ = 0.001 |
| 3270 reflections | $\Delta\rho_{max}$ = 0.14 e Å$^{-3}$ |
| 199 parameters | $\Delta\rho_{min}$ = −0.20 e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.06301 (7) | 0.63509 (6) | 0.91292 (14) | 0.0458 (3) |
| N1 | 0.07867 (7) | 0.52183 (7) | 0.88728 (15) | 0.0363 (4) |
| C1 | 0.14376 (10) | 0.52718 (10) | 0.9744 (2) | 0.0416 (5) |
| H1 | 0.1614 | 0.5668 | 1.0271 | 0.062* |
| C2 | 0.17911 (10) | 0.46711 (9) | 0.9736 (2) | 0.0449 (5) |
| H2 | 0.2250 | 0.4575 | 1.0246 | 0.067* |
| C3 | 0.13464 (10) | 0.42195 (9) | 0.8828 (2) | 0.0439 (5) |
| H3 | 0.1454 | 0.3761 | 0.8625 | 0.066* |
| C4 | 0.07299 (10) | 0.45515 (9) | 0.8283 (2) | 0.0376 (4) |
| C5 | 0.01475 (10) | 0.42109 (9) | 0.73953 (18) | 0.0385 (4) |
| H5 | 0.0220 | 0.3749 | 0.7156 | 0.058* |
| N2 | −0.04575 (8) | 0.44677 (7) | 0.68969 (16) | 0.0374 (4) |
| N3 | −0.01941 (7) | 0.57710 (7) | 0.75188 (16) | 0.0379 (4) |
| H3N | −0.0357 | 0.5364 | 0.7318 | 0.057* |
| C6 | −0.09948 (9) | 0.40501 (9) | 0.61349 (19) | 0.0356 (4) |
| C7 | −0.14260 (10) | 0.43399 (9) | 0.4869 (2) | 0.0407 (5) |
| H7 | −0.1360 | 0.4797 | 0.4575 | 0.061* |
| C8 | −0.19501 (10) | 0.39616 (9) | 0.4044 (2) | 0.0416 (5) |
| H8 | −0.2235 | 0.4157 | 0.3162 | 0.062* |
| C9 | −0.20604 (10) | 0.33021 (9) | 0.4496 (2) | 0.0447 (5) |
| H9 | −0.2422 | 0.3044 | 0.3927 | 0.067* |
| C10 | −0.16424 (10) | 0.30146 (9) | 0.5786 (2) | 0.0466 (5) |
| H10 | −0.1719 | 0.2560 | 0.6100 | 0.070* |
| C11 | −0.11140 (10) | 0.33934 (9) | 0.6612 (2) | 0.0415 (5) |
| H11 | −0.0834 | 0.3201 | 0.7506 | 0.062* |
| C12 | 0.03966 (10) | 0.58330 (9) | 0.8518 (2) | 0.0380 (4) |
| C13 | −0.05737 (10) | 0.63125 (8) | 0.67646 (19) | 0.0365 (4) |

| | | | | |
|---|---|---|---|---|
| C14 | −0.13024 (10) | 0.62156 (9) | 0.6346 (2) | 0.0440 (5) |
| H14 | −0.1543 | 0.5814 | 0.6670 | 0.066* |
| C15 | −0.16813 (11) | 0.67072 (9) | 0.5450 (2) | 0.0501 (5) |
| H15 | −0.2180 | 0.6640 | 0.5160 | 0.075* |
| C16 | −0.13333 (12) | 0.72922 (9) | 0.4984 (2) | 0.0507 (5) |
| H16 | −0.1590 | 0.7626 | 0.4361 | 0.076* |
| C17 | −0.06071 (11) | 0.73919 (9) | 0.5428 (2) | 0.0465 (5) |
| H17 | −0.0369 | 0.7797 | 0.5116 | 0.070* |
| C18 | −0.02248 (11) | 0.69054 (8) | 0.63223 (19) | 0.0410 (5) |
| H18 | 0.0271 | 0.6977 | 0.6630 | 0.061* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0529 (9) | 0.0375 (8) | 0.0470 (7) | −0.0021 (6) | −0.0039 (6) | −0.0077 (6) |
| N1 | 0.0378 (9) | 0.0349 (9) | 0.0362 (7) | −0.0004 (7) | 0.0002 (7) | −0.0013 (7) |
| C1 | 0.0363 (12) | 0.0492 (13) | 0.0393 (10) | −0.0057 (10) | −0.0025 (8) | −0.0016 (8) |
| C2 | 0.0409 (12) | 0.0450 (12) | 0.0489 (11) | 0.0015 (10) | −0.0008 (9) | 0.0034 (9) |
| C3 | 0.0466 (12) | 0.0370 (11) | 0.0480 (11) | 0.0026 (10) | 0.0026 (9) | −0.0003 (9) |
| C4 | 0.0405 (12) | 0.0360 (11) | 0.0362 (9) | −0.0023 (10) | 0.0037 (9) | 0.0006 (8) |
| C5 | 0.0470 (12) | 0.0343 (10) | 0.0342 (9) | −0.0039 (10) | 0.0070 (8) | −0.0009 (8) |
| N2 | 0.0385 (10) | 0.0370 (9) | 0.0369 (8) | −0.0013 (8) | 0.0025 (7) | 0.0001 (6) |
| N3 | 0.0394 (9) | 0.0315 (8) | 0.0428 (8) | −0.0016 (8) | −0.0021 (7) | −0.0018 (7) |
| C6 | 0.0389 (11) | 0.0332 (11) | 0.0346 (9) | −0.0028 (9) | 0.0037 (8) | −0.0026 (8) |
| C7 | 0.0444 (12) | 0.0361 (11) | 0.0415 (10) | −0.0021 (9) | 0.0068 (9) | 0.0028 (8) |
| C8 | 0.0400 (12) | 0.0472 (13) | 0.0377 (9) | −0.0008 (9) | 0.0022 (9) | 0.0018 (9) |
| C9 | 0.0445 (13) | 0.0473 (13) | 0.0423 (10) | −0.0082 (10) | 0.0017 (9) | −0.0043 (9) |
| C10 | 0.0550 (14) | 0.0397 (11) | 0.0450 (10) | −0.0087 (10) | 0.0007 (9) | 0.0019 (9) |
| C11 | 0.0461 (13) | 0.0391 (12) | 0.0394 (9) | −0.0039 (10) | 0.0013 (9) | −0.0001 (8) |
| C12 | 0.0412 (12) | 0.0369 (11) | 0.0358 (10) | −0.0005 (10) | 0.0053 (8) | −0.0004 (9) |
| C13 | 0.0447 (13) | 0.0302 (11) | 0.0345 (9) | 0.0038 (9) | 0.0046 (9) | −0.0020 (8) |
| C14 | 0.0431 (12) | 0.0393 (12) | 0.0497 (11) | −0.0012 (10) | 0.0004 (9) | 0.0020 (9) |
| C15 | 0.0503 (14) | 0.0427 (13) | 0.0573 (12) | 0.0033 (10) | −0.0048 (10) | 0.0028 (9) |
| C16 | 0.0669 (16) | 0.0387 (12) | 0.0465 (11) | 0.0071 (10) | −0.0047 (10) | 0.0030 (9) |
| C17 | 0.0648 (15) | 0.0333 (11) | 0.0415 (10) | −0.0014 (10) | 0.0027 (9) | 0.0005 (8) |
| C18 | 0.0496 (13) | 0.0389 (12) | 0.0344 (9) | −0.0027 (10) | 0.0031 (8) | −0.0045 (8) |

*Geometric parameters (Å, °)*

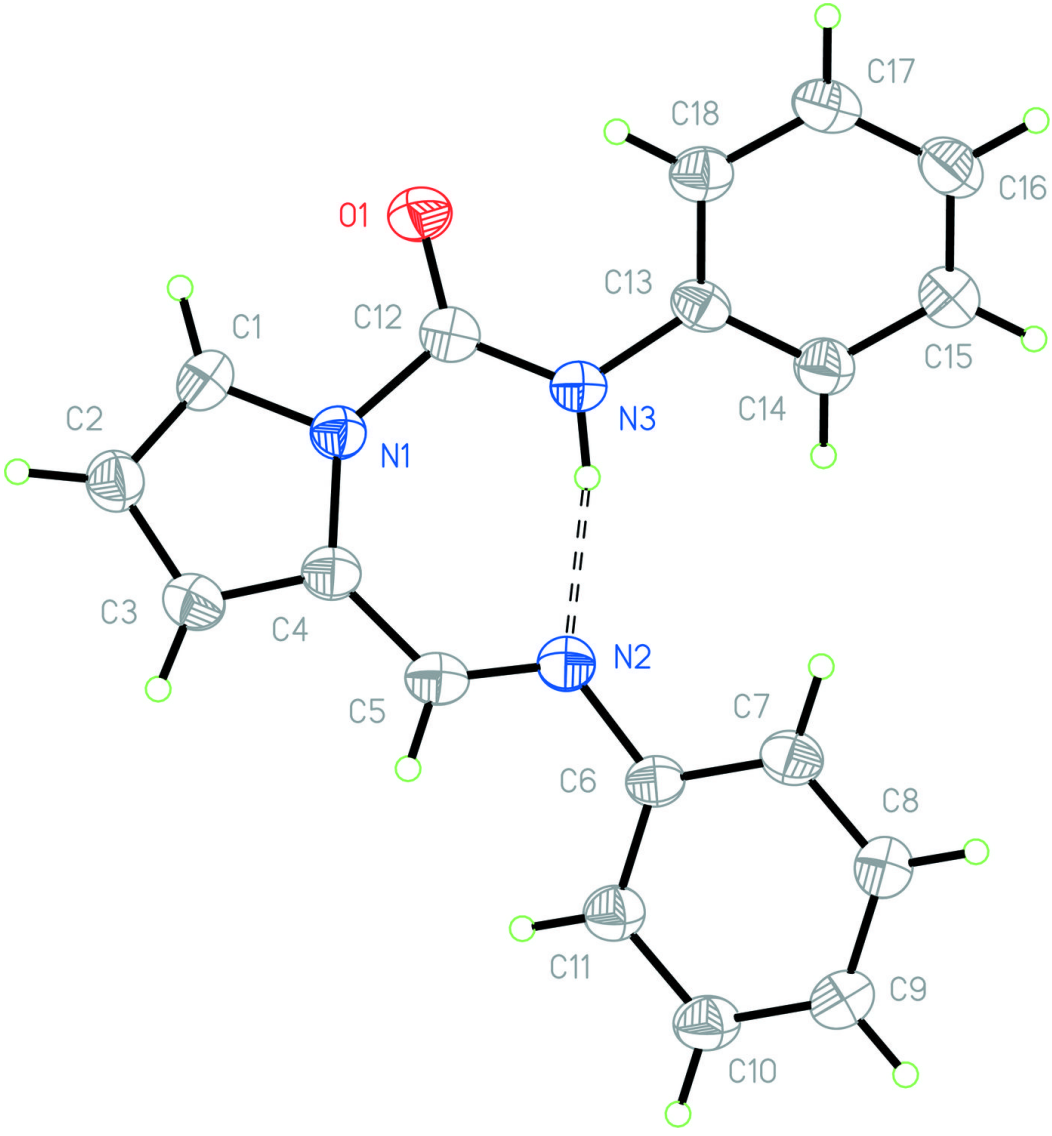
| | | | |
|---|---|---|---|
| O1—C12 | 1.2172 (19) | C7—H7 | 0.9500 |
| N1—C1 | 1.377 (2) | C8—C9 | 1.379 (2) |
| N1—C4 | 1.413 (2) | C8—H8 | 0.9500 |
| N1—C12 | 1.447 (2) | C9—C10 | 1.393 (2) |
| C1—C2 | 1.363 (2) | C9—H9 | 0.9500 |
| C1—H1 | 0.9500 | C10—C11 | 1.388 (2) |
| C2—C3 | 1.408 (2) | C10—H10 | 0.9500 |
| C2—H2 | 0.9500 | C11—H11 | 0.9500 |
| C3—C4 | 1.376 (2) | C13—C14 | 1.386 (2) |
| C3—H3 | 0.9500 | C13—C18 | 1.389 (2) |

| | | | |
|---|---|---|---|
| C4—C5 | 1.443 (2) | C14—C15 | 1.392 (2) |
| C5—N2 | 1.281 (2) | C14—H14 | 0.9500 |
| C5—H5 | 0.9500 | C15—C16 | 1.380 (2) |
| N2—C6 | 1.421 (2) | C15—H15 | 0.9500 |
| N3—C12 | 1.341 (2) | C16—C17 | 1.388 (2) |
| N3—C13 | 1.415 (2) | C16—H16 | 0.9500 |
| N3—H3N | 0.8800 | C17—C18 | 1.388 (2) |
| C6—C11 | 1.381 (2) | C17—H17 | 0.9500 |
| C6—C7 | 1.394 (2) | C18—H18 | 0.9500 |
| C7—C8 | 1.382 (2) | | |
| C1—N1—C4 | 107.41 (14) | C8—C9—C10 | 120.03 (17) |
| C1—N1—C12 | 117.15 (14) | C8—C9—H9 | 120.0 |
| C4—N1—C12 | 134.41 (14) | C10—C9—H9 | 120.0 |
| C2—C1—N1 | 109.87 (16) | C11—C10—C9 | 119.82 (17) |
| C2—C1—H1 | 125.1 | C11—C10—H10 | 120.1 |
| N1—C1—H1 | 125.1 | C9—C10—H10 | 120.1 |
| C1—C2—C3 | 106.96 (17) | C6—C11—C10 | 120.05 (17) |
| C1—C2—H2 | 126.5 | C6—C11—H11 | 120.0 |
| C3—C2—H2 | 126.5 | C10—C11—H11 | 120.0 |
| C4—C3—C2 | 108.79 (17) | O1—C12—N3 | 126.29 (17) |
| C4—C3—H3 | 125.6 | O1—C12—N1 | 118.13 (16) |
| C2—C3—H3 | 125.6 | N3—C12—N1 | 115.58 (16) |
| C3—C4—N1 | 106.96 (15) | C14—C13—C18 | 120.09 (17) |
| C3—C4—C5 | 121.87 (17) | C14—C13—N3 | 117.68 (15) |
| N1—C4—C5 | 131.01 (16) | C18—C13—N3 | 122.03 (16) |
| N2—C5—C4 | 126.63 (17) | C13—C14—C15 | 120.00 (18) |
| N2—C5—H5 | 116.7 | C13—C14—H14 | 120.0 |
| C4—C5—H5 | 116.7 | C15—C14—H14 | 120.0 |
| C5—N2—C6 | 119.43 (15) | C16—C15—C14 | 120.08 (19) |
| C12—N3—C13 | 124.72 (15) | C16—C15—H15 | 120.0 |
| C12—N3—H3N | 117.6 | C14—C15—H15 | 120.0 |
| C13—N3—H3N | 117.6 | C15—C16—C17 | 119.77 (18) |
| C11—C6—C7 | 119.87 (16) | C15—C16—H16 | 120.1 |
| C11—C6—N2 | 123.53 (15) | C17—C16—H16 | 120.1 |
| C7—C6—N2 | 116.59 (15) | C18—C17—C16 | 120.60 (18) |
| C8—C7—C6 | 119.98 (16) | C18—C17—H17 | 119.7 |
| C8—C7—H7 | 120.0 | C16—C17—H17 | 119.7 |
| C6—C7—H7 | 120.0 | C17—C18—C13 | 119.44 (18) |
| C9—C8—C7 | 120.20 (17) | C17—C18—H18 | 120.3 |
| C9—C8—H8 | 119.9 | C13—C18—H18 | 120.3 |
| C7—C8—H8 | 119.9 | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3N···N2 | 0.88 | 1.83 | 2.691 (2) | 166 |

**Fig. 1**


C17
C18
C16
O1
C13
C12
C1
C15
C2
N3
C14
N1
C3
C4
N2
C5
C7
C6
C8
C11
C9
C10