Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# (*E*)-*N*′-(4-Pyridylmethylene)-4-(8-quinolyloxy)butanohydrazide 0.25-hydrate

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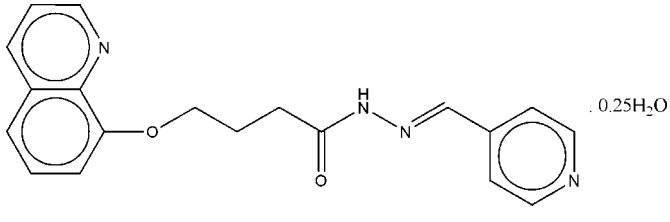
Received 6 January 2009; accepted 9 January 2009

Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.009 Å; disorder in solvent or counterion; $R$ factor = 0.058; $wR$ factor = 0.162; data-to-parameter ratio = 6.9.

The asymmetric unit of the title compound, $C_{19}H_{18}N_4O_2\cdot 0.25H_2O$, contains two organic molecules and a solvent water molecule with 50% occupancy. The two molecules differ in their conformations: in one molecule it is (+)*gauche-trans-trans*-(+)*gauche-trans*, whereas in the other it is (−)*gauche-trans-trans*-(−)*gauche-trans*. The dihedral angles between the pyridine ring and the quinoline ring system are 67.4 (3) and 68.0 (2)°. Molecules are linked into a supramolecular two-dimensional array *via* N—H···N hydrogen bonds, with each partially occupied water molecule connected *via* an O—H···O hydrogen bond. C—H···O interactions are also present.

## Related literature

For general background, see: Cai *et al.* (2003); Chen *et al.* (2005); Park *et al.* (2006); Karmakar *et al.* (2007). For related structures, see: Zheng *et al.* (2006, 2007, 2008); Xie *et al.* (2008).




## Experimental

### Crystal data

$C_{19}H_{18}N_4O_2\cdot 0.25H_2O$
$M_r$ = 1355.51
Monoclinic, $P2_1$
$a$ = 8.8816 (13) Å
$b$ = 17.420 (2) Å
$c$ = 11.3624 (17) Å
$\beta$ = 100.765 (3)°
$V$ = 1727.0 (4) Å$^3$
$Z$ = 1
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 295 K
0.32 × 0.26 × 0.22 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.972, $T_{max}$ = 0.981
9142 measured reflections
3169 independent reflections
1927 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.050

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.162
$S$ = 1.01
3169 reflections
460 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C22—H22···O4$^{i}$ | 0.93 | 2.51 | 3.224 (7) | 134 |
| C7—H7···N8$^{ii}$ | 0.93 | 2.55 | 3.466 (9) | 167 |
| C3—H3···O2$^{iii}$ | 0.93 | 2.52 | 3.353 (7) | 150 |
| C2—H1···N4$^{iv}$ | 0.93 | 2.55 | 3.390 (9) | 150 |
| O5—H39···O4$^{v}$ | 0.85 | 2.17 | 2.964 (10) | 156 |
| N6—H6···N1$^{iii}$ | 0.86 | 2.10 | 2.934 (6) | 163 |
| N2—H2···N5$^{i}$ | 0.86 | 2.25 | 3.077 (7) | 161 |

Symmetry codes: (i) $x, y, z-1$; (ii) $-x+3, y-\frac{1}{2}, -z+2$; (iii) $x, y, z+1$; (iv) $-x+1, y+\frac{1}{2}, -z+1$; (v) $-x+1, y+\frac{1}{2}, -z+2$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This work was supported by grants from Qinzhou University Foundation and the Young Key Teacher's Plan of Guangxi Zhuang Autonomous Region of the People's Republic of China [grant Nos. 2008XJKY-10B and 2008(30)].

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2355).

## References

Bruker (2007). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cai, Y. P., Zhang, L., Shi, J. L., Zhang, H. X. & Kang, B. S. (2003). *Chin. J. Struct. Chem.* **22**, 587–590.
Chen, C. L., Goforth, A. M., Smith, M. D., Gemmill, W. R., Su, C. Y. & Loye, H. C. (2005). *J. Cluster Sci.*, 16, 477–487.
Karmakar, A., Sarma, R. J. & Baruah, J. B. (2007). *CrystEngComm*, **9**, 379–389.
Park, K. M., Moon, S. T., Kang, Y. J., Kim, H. J., Seo, J. & Lee, S. S. (2006). *Inorg. Chem. Commun.* **9**, 671–674.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Xie, H., Meng, S.-M., Fan, Y.-Q. & Yang, G.-C. (2008). *Acta Cryst.* E**64**, o2114.
Zheng, Z.-B., Li, J.-K., Sun, Y.-F. & Wu, R.-T. (2008). *Acta Cryst.* E**64**, o297.
Zheng, Z.-B., Li, J.-K., Wu, R.-T. & Sun, Y.-F. (2006). *Acta Cryst.* E**62**, o4611–o4612.
Zheng, Z.-B., Wu, R.-T., Li, J.-K. & Lu, J.-R. (2007). *Acta Cryst.* E**63**, o3284.

# supplementary materials

*Acta Cryst.* (2009). E**65**, o295 [ **doi:10.1107/S160053680900110X** ]

# (*E*)-*N*'-(4-Pyridylmethylene)-4-(8-quinolyloxy)butanohydrazide 0.25-hydrate

**M.-E. Chen and J.-M. Li**

## Comment

The coordination chemistry of 8-hydroxyquinoline and its derivatives is well studied (Cai *et al.*, 2003; Chen *et al.*, 2005; Park *et al.*, 2006; Karmakar *et al.* 2007). In the course of our studies searching for good extractants of metal ions, the title compound, (I), was synthesized and its crystal structure determined. The asymmetric unit comprises two independent molecules which differ in conformation and a water molecule with a 50% site occupancy factor. In one molecule, the conformation along the O1—C10—C11—C12—C13—N2—N3—C14 bond sequence is (+)*gauche*-*trans*-*trans*-(+)*gauche*-*trans*, whereas in the second molecule the conformation is (-)*gauche*-*trans*-*trans*-(-)*gauche*-*trans* along the corresponding O3—C29—C30—C31—C32—N6—N7—C33 bond sequence. Despite these differences, the dihedral angles between the mean planes of the pyridine and quinoline rings are not very different, i.e. 67.4 (3)° and 68.0 (2)° for the molecules containing atoms O1 and O3, respectively. The two independent molecules are linked to a supermolecular 2D array *via* N—H···N hydrogen bonds supported by C—H···O contacts, Table 1. Each partially occupied water molecules is connected to a layer via a O—H···O hydrogen bond.

## Experimental

4-(Quinolin-8-yloxy)butanohydrazide (0.01 mol), 4-pyridylaldehyde (0.01 mol), ethanol (40 ml) and some drops of acetic acid were added to a 100 ml flask, and refluxed for 8 h. After cooling to room temperature, the mixture was filtered. Colourless crystals were obtained by slow evaporation of a tetrahydrofuran solution over a period of 2 days; m.p. 438 K. Analysis found: C 67.34, H 5.50, N 16.53; $C_{76}H_{74}N_{16}O_9$ requires: C 67.30, H 5.54, N 16.51.

## Refinement

All H atoms were placed in idealized positions (C—H = 0.93–0.97 Å, N—H = 0.86 Å and O—H = 0.85 Å) and refined in the riding model approximation with $U_{iso}$(H) = 1.2$U_{eq}$(C or N) and $U_{iso}$(H) = 1.5$U_{eq}$(O). The water molecule was included in the model with a 50% site occupancy factor based on elemental analysis and refinement. In the absence of significant anomalous scattering effects, X Friedel pairs were averaged in the final refinement.

## Figures

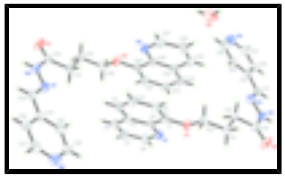


Fig. 1. The molecular structures of the two independent molecules and the water molecule of crystallization (50% site occupancy) in (I), showing atom numbering scheme and displacement ellipsoids at the 30% probability level.

## (*E*)-*N*′-(4-Pyridylmethylene)-4-(8-quinolyloxy)butanohydrazide 0.25-hydrate

*Crystal data*

| | |
|---|---|
| $C_{19}H_{18}N_4O_2 \cdot 0.25H_2O$ | $F_{000} = 714$ |
| $M_r = 1355.51$ | $D_x = 1.303$ Mg m$^{-3}$ |
| Monoclinic, $P2_1$ | Melting point: 438 K |
| Hall symbol: P 2yb | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 8.8816$ (13) Å | Cell parameters from 1165 reflections |
| $b = 17.420$ (2) Å | $\theta = 2.2$–$20.0$º |
| $c = 11.3624$ (17) Å | $\mu = 0.09$ mm$^{-1}$ |
| $\beta = 100.765$ (3)º | $T = 295$ K |
| $V = 1727.0$ (4) Å$^3$ | Block, colorless |
| $Z = 1$ | 0.32 × 0.26 × 0.22 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 3169 independent reflections |
| Radiation source: fine-focus sealed tube | 1927 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.050$ |
| $T = 295$ K | $\theta_{max} = 25.1$º |
| φ and ω scans | $\theta_{min} = 1.8$º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -10 \rightarrow 10$ |
| $T_{min} = 0.972$, $T_{max} = 0.981$ | $k = -13 \rightarrow 20$ |
| 9142 measured reflections | $l = -13 \rightarrow 13$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.058$ | $w = 1/[\sigma^2(F_o^2) + (0.0877P)^2 + 0.001P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.162$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S = 1.01$ | $\Delta\rho_{max} = 0.32$ e Å$^{-3}$ |
| 3169 reflections | $\Delta\rho_{min} = -0.21$ e Å$^{-3}$ |
| 460 parameters | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | Absolute structure: nd |
| Secondary atom site location: difference Fourier map | |

*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 $F^2 > \sigma(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| N1 | 1.1571 (5) | 0.0923 (3) | 0.6124 (4) | 0.0478 (12) | |
| N2 | 0.7191 (6) | 0.1522 (3) | 0.1045 (4) | 0.0566 (14) | |
| H2 | 0.7211 | 0.1460 | 0.0297 | 0.068* | |
| N3 | 0.6437 (6) | 0.1008 (3) | 0.1636 (4) | 0.0510 (13) | |
| N4 | 0.3307 (7) | −0.1253 (4) | 0.2546 (6) | 0.0817 (18) | |
| N5 | 0.6503 (5) | 0.1139 (3) | 0.8355 (4) | 0.0495 (12) | |
| N6 | 1.0930 (6) | 0.0735 (3) | 1.3512 (4) | 0.0510 (13) | |
| H6 | 1.0909 | 0.0810 | 1.4257 | 0.061* | |
| N7 | 1.1691 (6) | 0.1246 (3) | 1.2916 (4) | 0.0474 (12) | |
| N8 | 1.4779 (7) | 0.3522 (3) | 1.1941 (6) | 0.0674 (15) | |
| O1 | 0.9800 (4) | 0.2139 (2) | 0.5363 (3) | 0.0497 (10) | |
| O2 | 0.8522 (6) | 0.2626 (3) | 0.1125 (4) | 0.0769 (14) | |
| O3 | 0.8302 (5) | −0.0034 (2) | 0.9236 (3) | 0.0511 (10) | |
| O4 | 0.9623 (6) | −0.0348 (2) | 1.3516 (4) | 0.0707 (14) | |
| O5 | 0.2645 (9) | 0.3398 (5) | 0.6539 (8) | 0.072 (3) | 0.50 |
| H39 | 0.2124 | 0.3805 | 0.6357 | 0.107* | 0.50 |
| H40 | 0.2794 | 0.3362 | 0.7297 | 0.107* | 0.50 |
| C1 | 1.0067 (7) | 0.1999 (3) | 0.6560 (5) | 0.0430 (14) | |
| C2 | 0.9470 (7) | 0.2422 (3) | 0.7384 (5) | 0.0491 (15) | |
| H1 | 0.8840 | 0.2840 | 0.7134 | 0.059* | |
| C3 | 0.9807 (7) | 0.2225 (4) | 0.8599 (5) | 0.0557 (16) | |
| H3 | 0.9389 | 0.2514 | 0.9147 | 0.067* | |
| C4 | 1.0719 (7) | 0.1628 (4) | 0.8992 (5) | 0.0569 (17) | |
| H4 | 1.0932 | 0.1515 | 0.9806 | 0.068* | |
| C5 | 1.1361 (7) | 0.1168 (3) | 0.8187 (5) | 0.0478 (15) | |
| C6 | 1.2298 (7) | 0.0529 (4) | 0.8537 (5) | 0.0583 (17) | |
| H5 | 1.2534 | 0.0389 | 0.9340 | 0.070* | |
| C7 | 1.2853 (8) | 0.0120 (4) | 0.7713 (6) | 0.0662 (18) | |
| H7 | 1.3495 | −0.0298 | 0.7938 | 0.079* | |
| C8 | 1.2449 (8) | 0.0333 (4) | 0.6505 (6) | 0.0608 (17) | |
| H8 | 1.2824 | 0.0039 | 0.5940 | 0.073* | |
| C9 | 1.1020 (6) | 0.1352 (3) | 0.6951 (4) | 0.0391 (13) | |
| C10 | 0.8914 (7) | 0.2810 (3) | 0.4948 (5) | 0.0508 (15) | |

| | | | | |
|---|---|---|---|---|
| H10A | 0.9416 | 0.3268 | 0.5318 | 0.061* |
| H10B | 0.7903 | 0.2776 | 0.5152 | 0.061* |
| C11 | 0.8788 (7) | 0.2845 (3) | 0.3596 (5) | 0.0537 (16) |
| H11A | 0.8270 | 0.3315 | 0.3295 | 0.064* |
| H11B | 0.9808 | 0.2854 | 0.3406 | 0.064* |
| C12 | 0.7917 (7) | 0.2168 (3) | 0.2986 (4) | 0.0498 (15) |
| H12A | 0.6867 | 0.2192 | 0.3110 | 0.060* |
| H12B | 0.8370 | 0.1699 | 0.3353 | 0.060* |
| C13 | 0.7914 (7) | 0.2139 (4) | 0.1658 (5) | 0.0517 (15) |
| C14 | 0.5817 (7) | 0.0439 (4) | 0.1021 (5) | 0.0523 (15) |
| H14 | 0.5915 | 0.0389 | 0.0224 | 0.063* |
| C15 | 0.4960 (7) | −0.0130 (3) | 0.1556 (5) | 0.0491 (15) |
| C16 | 0.4348 (7) | −0.0766 (4) | 0.0895 (6) | 0.0593 (16) |
| H16 | 0.4488 | −0.0832 | 0.0110 | 0.071* |
| C17 | 0.3533 (8) | −0.1299 (4) | 0.1419 (7) | 0.075 (2) |
| H17 | 0.3116 | −0.1715 | 0.0960 | 0.090* |
| C18 | 0.3892 (9) | −0.0646 (5) | 0.3151 (7) | 0.076 (2) |
| H18 | 0.3749 | −0.0600 | 0.3938 | 0.091* |
| C19 | 0.4703 (7) | −0.0070 (4) | 0.2711 (5) | 0.0581 (17) |
| H19 | 0.5068 | 0.0350 | 0.3186 | 0.070* |
| C20 | 0.8106 (7) | 0.0064 (3) | 0.8035 (5) | 0.0442 (14) |
| C21 | 0.8736 (7) | −0.0391 (4) | 0.7273 (5) | 0.0558 (16) |
| H21 | 0.9340 | −0.0807 | 0.7577 | 0.067* |
| C22 | 0.8485 (8) | −0.0239 (4) | 0.6043 (6) | 0.0649 (19) |
| H22 | 0.8948 | −0.0546 | 0.5543 | 0.078* |
| C23 | 0.7566 (8) | 0.0355 (4) | 0.5573 (5) | 0.0649 (19) |
| H23 | 0.7396 | 0.0446 | 0.4752 | 0.078* |
| C24 | 0.6872 (7) | 0.0832 (4) | 0.6320 (5) | 0.0512 (16) |
| C25 | 0.5906 (8) | 0.1436 (4) | 0.5884 (6) | 0.070 (2) |
| H25 | 0.5701 | 0.1542 | 0.5068 | 0.084* |
| C26 | 0.5250 (8) | 0.1879 (4) | 0.6658 (6) | 0.070 (2) |
| H26 | 0.4589 | 0.2280 | 0.6378 | 0.083* |
| C27 | 0.5617 (8) | 0.1702 (4) | 0.7890 (6) | 0.0663 (18) |
| H27 | 0.5194 | 0.2010 | 0.8412 | 0.080* |
| C28 | 0.7132 (7) | 0.0686 (3) | 0.7572 (5) | 0.0455 (14) |
| C29 | 0.9213 (7) | −0.0686 (3) | 0.9732 (5) | 0.0523 (15) |
| H29A | 1.0227 | −0.0659 | 0.9533 | 0.063* |
| H29B | 0.8731 | −0.1161 | 0.9414 | 0.063* |
| C30 | 0.9317 (7) | −0.0652 (3) | 1.1079 (5) | 0.0472 (14) |
| H30A | 0.8291 | −0.0649 | 1.1259 | 0.057* |
| H30B | 0.9839 | −0.1107 | 1.1441 | 0.057* |
| C31 | 1.0176 (7) | 0.0060 (3) | 1.1622 (5) | 0.0479 (14) |
| H31A | 1.1217 | 0.0044 | 1.1477 | 0.057* |
| H31B | 0.9684 | 0.0514 | 1.1232 | 0.057* |
| C32 | 1.0214 (7) | 0.0116 (3) | 1.2951 (5) | 0.0492 (14) |
| C33 | 1.2327 (7) | 0.1813 (4) | 1.3530 (5) | 0.0517 (15) |
| H33 | 1.2256 | 0.1860 | 1.4333 | 0.062* |
| C34 | 1.3162 (6) | 0.2387 (3) | 1.2971 (5) | 0.0448 (14) |
| C35 | 1.3416 (7) | 0.2328 (4) | 1.1804 (5) | 0.0580 (17) |

| | | | | |
|---|---|---|---|---|
| H35 | 1.3053 | 0.1904 | 1.1339 | 0.070* |
| C36 | 1.4204 (8) | 0.2895 (4) | 1.1339 (6) | 0.071 (2) |
| H36 | 1.4347 | 0.2840 | 1.0553 | 0.085* |
| C37 | 1.4515 (8) | 0.3568 (4) | 1.3043 (6) | 0.0665 (19) |
| H37 | 1.4879 | 0.4002 | 1.3482 | 0.080* |
| C38 | 1.3756 (7) | 0.3035 (4) | 1.3600 (6) | 0.0603 (17) |
| H38 | 1.3640 | 0.3107 | 1.4389 | 0.072* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.053 (3) | 0.049 (3) | 0.043 (3) | 0.012 (2) | 0.012 (2) | 0.000 (2) |
| N2 | 0.068 (4) | 0.063 (4) | 0.042 (3) | −0.004 (3) | 0.017 (3) | 0.005 (2) |
| N3 | 0.052 (3) | 0.061 (3) | 0.041 (3) | 0.000 (3) | 0.010 (2) | 0.001 (3) |
| N4 | 0.084 (5) | 0.072 (5) | 0.091 (5) | −0.008 (4) | 0.020 (4) | 0.010 (4) |
| N5 | 0.051 (3) | 0.052 (3) | 0.045 (3) | −0.006 (3) | 0.005 (2) | 0.000 (2) |
| N6 | 0.070 (4) | 0.050 (3) | 0.036 (3) | −0.004 (3) | 0.019 (3) | 0.003 (2) |
| N7 | 0.056 (3) | 0.042 (3) | 0.045 (3) | 0.000 (2) | 0.012 (2) | 0.004 (2) |
| N8 | 0.069 (4) | 0.059 (4) | 0.075 (4) | −0.009 (3) | 0.016 (3) | 0.007 (3) |
| O1 | 0.067 (3) | 0.046 (2) | 0.036 (2) | 0.013 (2) | 0.0091 (18) | 0.0019 (17) |
| O2 | 0.110 (4) | 0.072 (3) | 0.055 (3) | −0.020 (3) | 0.031 (3) | 0.010 (2) |
| O3 | 0.066 (3) | 0.054 (3) | 0.033 (2) | 0.010 (2) | 0.0065 (18) | 0.0019 (17) |
| O4 | 0.111 (4) | 0.060 (3) | 0.048 (2) | −0.026 (3) | 0.032 (3) | 0.002 (2) |
| O5 | 0.059 (6) | 0.055 (5) | 0.102 (7) | 0.002 (4) | 0.018 (5) | −0.020 (5) |
| C1 | 0.057 (4) | 0.037 (3) | 0.035 (3) | −0.005 (3) | 0.009 (3) | −0.001 (2) |
| C2 | 0.063 (4) | 0.041 (3) | 0.047 (4) | −0.002 (3) | 0.017 (3) | −0.011 (3) |
| C3 | 0.073 (4) | 0.055 (4) | 0.043 (3) | −0.006 (4) | 0.021 (3) | −0.013 (3) |
| C4 | 0.068 (4) | 0.069 (5) | 0.031 (3) | −0.019 (4) | 0.002 (3) | −0.004 (3) |
| C5 | 0.049 (4) | 0.050 (4) | 0.041 (3) | 0.000 (3) | 0.000 (3) | −0.001 (3) |
| C6 | 0.057 (4) | 0.074 (5) | 0.039 (4) | 0.003 (4) | −0.003 (3) | 0.009 (3) |
| C7 | 0.064 (4) | 0.070 (5) | 0.062 (4) | 0.019 (4) | 0.004 (3) | 0.014 (4) |
| C8 | 0.069 (4) | 0.058 (4) | 0.058 (4) | 0.016 (4) | 0.018 (3) | 0.002 (3) |
| C9 | 0.046 (3) | 0.037 (3) | 0.034 (3) | −0.001 (3) | 0.006 (2) | 0.000 (2) |
| C10 | 0.061 (4) | 0.039 (3) | 0.050 (4) | 0.003 (3) | 0.004 (3) | −0.002 (3) |
| C11 | 0.065 (4) | 0.049 (4) | 0.047 (4) | 0.001 (3) | 0.010 (3) | 0.006 (3) |
| C12 | 0.062 (4) | 0.051 (4) | 0.036 (3) | 0.004 (3) | 0.007 (3) | 0.008 (3) |
| C13 | 0.057 (4) | 0.054 (4) | 0.044 (3) | 0.002 (3) | 0.011 (3) | 0.007 (3) |
| C14 | 0.060 (4) | 0.059 (4) | 0.039 (3) | 0.010 (3) | 0.013 (3) | 0.002 (3) |
| C15 | 0.046 (4) | 0.049 (4) | 0.049 (4) | 0.003 (3) | 0.003 (3) | 0.004 (3) |
| C16 | 0.068 (4) | 0.048 (4) | 0.061 (4) | 0.009 (3) | 0.009 (3) | −0.006 (3) |
| C17 | 0.078 (5) | 0.052 (5) | 0.093 (6) | −0.003 (4) | 0.007 (4) | −0.006 (4) |
| C18 | 0.075 (5) | 0.084 (6) | 0.069 (5) | 0.006 (5) | 0.017 (4) | 0.006 (4) |
| C19 | 0.068 (4) | 0.060 (4) | 0.046 (4) | −0.003 (4) | 0.009 (3) | 0.003 (3) |
| C20 | 0.051 (3) | 0.048 (3) | 0.033 (3) | −0.006 (3) | 0.003 (3) | −0.004 (3) |
| C21 | 0.063 (4) | 0.064 (4) | 0.041 (3) | −0.003 (3) | 0.010 (3) | −0.011 (3) |
| C22 | 0.072 (5) | 0.077 (5) | 0.050 (4) | −0.019 (4) | 0.023 (3) | −0.019 (4) |
| C23 | 0.072 (4) | 0.087 (5) | 0.036 (3) | −0.023 (4) | 0.010 (3) | −0.004 (4) |
| C24 | 0.053 (4) | 0.062 (4) | 0.037 (3) | −0.021 (3) | 0.005 (3) | 0.004 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C25 | 0.071 (5) | 0.080 (5) | 0.053 (4) | −0.017 (4) | −0.004 (4) | 0.015 (4) |
| C26 | 0.065 (5) | 0.065 (5) | 0.070 (5) | −0.006 (4) | −0.009 (4) | 0.020 (4) |
| C27 | 0.067 (5) | 0.060 (4) | 0.071 (5) | 0.007 (4) | 0.013 (4) | 0.006 (4) |
| C28 | 0.046 (3) | 0.055 (4) | 0.035 (3) | −0.013 (3) | 0.008 (3) | −0.006 (3) |
| C29 | 0.067 (4) | 0.040 (3) | 0.048 (3) | 0.001 (3) | 0.005 (3) | −0.002 (3) |
| C30 | 0.058 (4) | 0.036 (3) | 0.045 (3) | 0.003 (3) | 0.004 (3) | 0.010 (3) |
| C31 | 0.058 (4) | 0.044 (3) | 0.042 (3) | −0.004 (3) | 0.010 (3) | 0.007 (3) |
| C32 | 0.064 (4) | 0.045 (3) | 0.040 (3) | −0.001 (3) | 0.013 (3) | 0.002 (3) |
| C33 | 0.054 (4) | 0.055 (4) | 0.045 (4) | −0.001 (3) | 0.005 (3) | −0.001 (3) |
| C34 | 0.047 (3) | 0.039 (3) | 0.049 (4) | 0.005 (3) | 0.011 (3) | 0.000 (3) |
| C35 | 0.060 (4) | 0.056 (4) | 0.057 (4) | −0.011 (3) | 0.007 (3) | −0.009 (3) |
| C36 | 0.079 (5) | 0.080 (5) | 0.057 (4) | −0.018 (4) | 0.023 (4) | 0.004 (4) |
| C37 | 0.071 (5) | 0.050 (4) | 0.075 (5) | −0.005 (4) | 0.005 (4) | −0.008 (4) |
| C38 | 0.062 (4) | 0.060 (4) | 0.059 (4) | −0.002 (4) | 0.012 (3) | 0.000 (3) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| N1—C8 | 1.314 (7) | C12—H12A | 0.9700 |
| N1—C9 | 1.361 (6) | C12—H12B | 0.9700 |
| N2—C13 | 1.373 (8) | C14—C15 | 1.451 (8) |
| N2—N3 | 1.368 (6) | C14—H14 | 0.9300 |
| N2—H2 | 0.8600 | C15—C19 | 1.378 (8) |
| N3—C14 | 1.277 (7) | C15—C16 | 1.391 (8) |
| N4—C18 | 1.314 (9) | C16—C17 | 1.378 (9) |
| N4—C17 | 1.335 (9) | C16—H16 | 0.9300 |
| N5—C27 | 1.306 (8) | C17—H17 | 0.9300 |
| N5—C28 | 1.381 (7) | C18—C19 | 1.382 (9) |
| N6—C32 | 1.349 (7) | C18—H18 | 0.9300 |
| N6—N7 | 1.371 (6) | C19—H19 | 0.9300 |
| N6—H6 | 0.8600 | C20—C21 | 1.368 (7) |
| N7—C33 | 1.278 (7) | C20—C28 | 1.425 (8) |
| N8—C37 | 1.317 (8) | C21—C22 | 1.398 (8) |
| N8—C36 | 1.339 (9) | C21—H21 | 0.9300 |
| O1—C1 | 1.359 (6) | C22—C23 | 1.364 (9) |
| O1—C10 | 1.438 (6) | C22—H22 | 0.9300 |
| O2—C13 | 1.224 (7) | C23—C24 | 1.411 (8) |
| O3—C20 | 1.354 (6) | C23—H23 | 0.9300 |
| O3—C29 | 1.445 (7) | C24—C25 | 1.389 (9) |
| O4—C32 | 1.212 (7) | C24—C28 | 1.421 (7) |
| O5—H39 | 0.8500 | C25—C26 | 1.377 (9) |
| O5—H40 | 0.8499 | C25—H25 | 0.9300 |
| C1—C2 | 1.373 (7) | C26—C27 | 1.410 (9) |
| C1—C9 | 1.429 (7) | C26—H26 | 0.9300 |
| C2—C3 | 1.400 (8) | C27—H27 | 0.9300 |
| C2—H1 | 0.9300 | C29—C30 | 1.518 (7) |
| C3—C4 | 1.342 (9) | C29—H29A | 0.9700 |
| C3—H3 | 0.9300 | C29—H29B | 0.9700 |
| C4—C5 | 1.414 (8) | C30—C31 | 1.524 (8) |
| C4—H4 | 0.9300 | C30—H30A | 0.9700 |

| | | | |
|---|---|---|---|
| C5—C6 | 1.402 (8) | C30—H30B | 0.9700 |
| C5—C9 | 1.417 (7) | C31—C32 | 1.507 (8) |
| C6—C7 | 1.343 (9) | C31—H31A | 0.9700 |
| C6—H5 | 0.9300 | C31—H31B | 0.9700 |
| C7—C8 | 1.402 (9) | C33—C34 | 1.460 (8) |
| C7—H7 | 0.9300 | C33—H33 | 0.9300 |
| C8—H8 | 0.9300 | C34—C38 | 1.386 (8) |
| C10—C11 | 1.521 (7) | C34—C35 | 1.389 (8) |
| C10—H10A | 0.9700 | C35—C36 | 1.373 (8) |
| C10—H10B | 0.9700 | C35—H35 | 0.9300 |
| C11—C12 | 1.506 (8) | C36—H36 | 0.9300 |
| C11—H11A | 0.9700 | C37—C38 | 1.370 (9) |
| C11—H11B | 0.9700 | C37—H37 | 0.9300 |
| C12—C13 | 1.509 (7) | C38—H38 | 0.9300 |
| C8—N1—C9 | 118.0 (5) | C16—C17—H17 | 118.0 |
| C13—N2—N3 | 119.3 (5) | N4—C18—C19 | 125.2 (7) |
| C13—N2—H2 | 120.4 | N4—C18—H18 | 117.4 |
| N3—N2—H2 | 120.4 | C19—C18—H18 | 117.4 |
| C14—N3—N2 | 116.1 (5) | C15—C19—C18 | 118.6 (6) |
| C18—N4—C17 | 115.7 (6) | C15—C19—H19 | 120.7 |
| C27—N5—C28 | 117.0 (5) | C18—C19—H19 | 120.7 |
| C32—N6—N7 | 121.2 (4) | O3—C20—C21 | 125.0 (5) |
| C32—N6—H6 | 119.4 | O3—C20—C28 | 115.0 (5) |
| N7—N6—H6 | 119.4 | C21—C20—C28 | 119.9 (5) |
| C33—N7—N6 | 116.2 (5) | C20—C21—C22 | 121.0 (6) |
| C37—N8—C36 | 114.6 (6) | C20—C21—H21 | 119.5 |
| C1—O1—C10 | 117.2 (4) | C22—C21—H21 | 119.5 |
| C20—O3—C29 | 116.7 (4) | C23—C22—C21 | 120.5 (6) |
| H39—O5—H40 | 106.4 | C23—C22—H22 | 119.8 |
| O1—C1—C2 | 124.6 (5) | C21—C22—H22 | 119.8 |
| O1—C1—C9 | 115.8 (4) | C22—C23—C24 | 120.6 (6) |
| C2—C1—C9 | 119.5 (5) | C22—C23—H23 | 119.7 |
| C1—C2—C3 | 120.2 (6) | C24—C23—H23 | 119.7 |
| C1—C2—H1 | 119.9 | C25—C24—C23 | 122.8 (6) |
| C3—C2—H1 | 119.9 | C25—C24—C28 | 117.8 (6) |
| C4—C3—C2 | 121.4 (5) | C23—C24—C28 | 119.3 (6) |
| C4—C3—H3 | 119.3 | C24—C25—C26 | 120.2 (6) |
| C2—C3—H3 | 119.3 | C24—C25—H25 | 119.9 |
| C3—C4—C5 | 121.1 (6) | C26—C25—H25 | 119.9 |
| C3—C4—H4 | 119.5 | C25—C26—C27 | 117.7 (7) |
| C5—C4—H4 | 119.5 | C25—C26—H26 | 121.2 |
| C6—C5—C9 | 117.8 (5) | C27—C26—H26 | 121.2 |
| C6—C5—C4 | 123.8 (6) | N5—C27—C26 | 125.2 (7) |
| C9—C5—C4 | 118.4 (5) | N5—C27—H27 | 117.4 |
| C7—C6—C5 | 120.0 (6) | C26—C27—H27 | 117.4 |
| C7—C6—H5 | 120.0 | N5—C28—C24 | 122.2 (6) |
| C5—C6—H5 | 120.0 | N5—C28—C20 | 119.1 (5) |
| C6—C7—C8 | 118.8 (6) | C24—C28—C20 | 118.7 (5) |
| C6—C7—H7 | 120.6 | O3—C29—C30 | 106.6 (4) |

| | | | |
|---|---|---|---|
| C8—C7—H7 | 120.6 | O3—C29—H29A | 110.4 |
| N1—C8—C7 | 123.9 (6) | C30—C29—H29A | 110.4 |
| N1—C8—H8 | 118.1 | O3—C29—H29B | 110.4 |
| C7—C8—H8 | 118.1 | C30—C29—H29B | 110.4 |
| N1—C9—C5 | 121.5 (5) | H29A—C29—H29B | 108.6 |
| N1—C9—C1 | 119.2 (5) | C31—C30—C29 | 111.8 (5) |
| C5—C9—C1 | 119.4 (5) | C31—C30—H30A | 109.2 |
| O1—C10—C11 | 107.2 (4) | C29—C30—H30A | 109.2 |
| O1—C10—H10A | 110.3 | C31—C30—H30B | 109.2 |
| C11—C10—H10A | 110.3 | C29—C30—H30B | 109.2 |
| O1—C10—H10B | 110.3 | H30A—C30—H30B | 107.9 |
| C11—C10—H10B | 110.3 | C32—C31—C30 | 112.0 (5) |
| H10A—C10—H10B | 108.5 | C32—C31—H31A | 109.2 |
| C12—C11—C10 | 111.7 (5) | C30—C31—H31A | 109.2 |
| C12—C11—H11A | 109.3 | C32—C31—H31B | 109.2 |
| C10—C11—H11A | 109.3 | C30—C31—H31B | 109.2 |
| C12—C11—H11B | 109.3 | H31A—C31—H31B | 107.9 |
| C10—C11—H11B | 109.3 | O4—C32—N6 | 119.7 (5) |
| H11A—C11—H11B | 107.9 | O4—C32—C31 | 123.8 (6) |
| C11—C12—C13 | 113.1 (5) | N6—C32—C31 | 116.5 (5) |
| C11—C12—H12A | 109.0 | N7—C33—C34 | 120.0 (5) |
| C13—C12—H12A | 109.0 | N7—C33—H33 | 120.0 |
| C11—C12—H12B | 109.0 | C34—C33—H33 | 120.0 |
| C13—C12—H12B | 109.0 | C38—C34—C35 | 116.0 (5) |
| H12A—C12—H12B | 107.8 | C38—C34—C33 | 120.6 (5) |
| O2—C13—N2 | 120.0 (5) | C35—C34—C33 | 123.4 (5) |
| O2—C13—C12 | 123.5 (6) | C36—C35—C34 | 119.9 (6) |
| N2—C13—C12 | 116.5 (5) | C36—C35—H35 | 120.0 |
| N3—C14—C15 | 120.5 (5) | C34—C35—H35 | 120.0 |
| N3—C14—H14 | 119.8 | N8—C36—C35 | 124.3 (6) |
| C15—C14—H14 | 119.8 | N8—C36—H36 | 117.9 |
| C19—C15—C16 | 117.3 (6) | C35—C36—H36 | 117.9 |
| C19—C15—C14 | 122.7 (5) | N8—C37—C38 | 126.1 (6) |
| C16—C15—C14 | 120.0 (5) | N8—C37—H37 | 117.0 |
| C17—C16—C15 | 119.1 (6) | C38—C37—H37 | 117.0 |
| C17—C16—H16 | 120.5 | C37—C38—C34 | 119.0 (6) |
| C15—C16—H16 | 120.5 | C37—C38—H38 | 120.5 |
| N4—C17—C16 | 124.0 (7) | C34—C38—H38 | 120.5 |
| N4—C17—H17 | 118.0 | | |
| C13—N2—N3—C14 | −178.6 (5) | N4—C18—C19—C15 | −1.3 (11) |
| C32—N6—N7—C33 | 179.8 (5) | C29—O3—C20—C21 | −1.9 (8) |
| C10—O1—C1—C2 | 4.5 (8) | C29—O3—C20—C28 | 176.6 (5) |
| C10—O1—C1—C9 | −176.7 (5) | O3—C20—C21—C22 | −179.2 (6) |
| O1—C1—C2—C3 | 179.5 (5) | C28—C20—C21—C22 | 2.3 (8) |
| C9—C1—C2—C3 | 0.7 (8) | C20—C21—C22—C23 | −1.9 (9) |
| C1—C2—C3—C4 | 0.4 (9) | C21—C22—C23—C24 | 0.9 (9) |
| C2—C3—C4—C5 | −0.8 (9) | C22—C23—C24—C25 | −178.9 (6) |
| C3—C4—C5—C6 | −178.7 (6) | C22—C23—C24—C28 | −0.3 (9) |
| C3—C4—C5—C9 | 0.1 (9) | C23—C24—C25—C26 | 179.4 (6) |

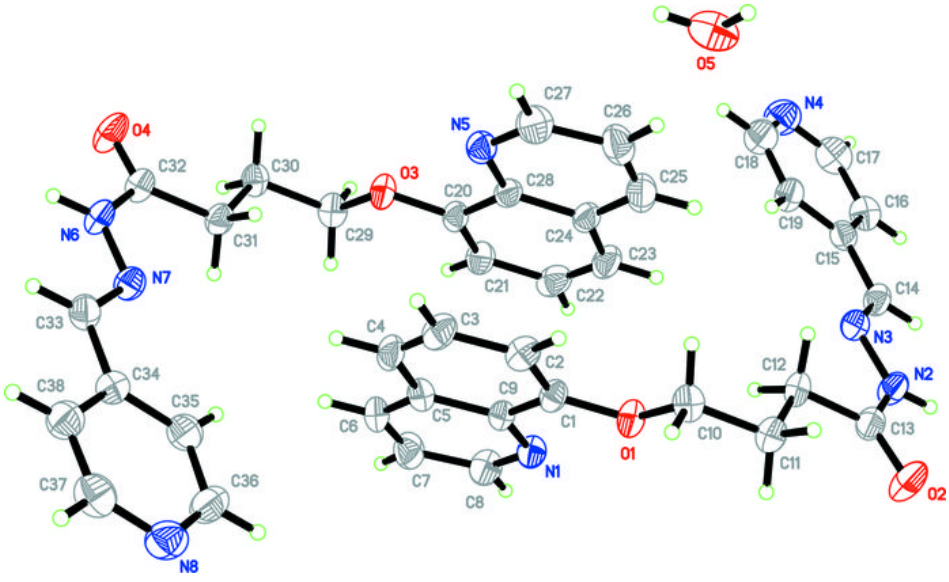
| | | | |
|---|---|---|---|
| C9—C5—C6—C7 | 0.8 (9) | C28—C24—C25—C26 | 0.9 (9) |
| C4—C5—C6—C7 | 179.6 (6) | C24—C25—C26—C27 | 1.0 (10) |
| C5—C6—C7—C8 | −1.5 (10) | C28—N5—C27—C26 | 0.3 (9) |
| C9—N1—C8—C7 | −0.1 (9) | C25—C26—C27—N5 | −1.7 (11) |
| C6—C7—C8—N1 | 1.3 (11) | C27—N5—C28—C24 | 1.7 (8) |
| C8—N1—C9—C5 | −0.7 (8) | C27—N5—C28—C20 | −179.9 (6) |
| C8—N1—C9—C1 | 179.8 (5) | C25—C24—C28—N5 | −2.3 (8) |
| C6—C5—C9—N1 | 0.4 (8) | C23—C24—C28—N5 | 179.1 (5) |
| C4—C5—C9—N1 | −178.5 (5) | C25—C24—C28—C20 | 179.3 (5) |
| C6—C5—C9—C1 | 179.9 (5) | C23—C24—C28—C20 | 0.7 (8) |
| C4—C5—C9—C1 | 1.0 (8) | O3—C20—C28—N5 | 1.2 (7) |
| O1—C1—C9—N1 | −0.8 (7) | C21—C20—C28—N5 | 179.9 (5) |
| C2—C1—C9—N1 | 178.1 (5) | O3—C20—C28—C24 | 179.6 (5) |
| O1—C1—C9—C5 | 179.7 (5) | C21—C20—C28—C24 | −1.7 (8) |
| C2—C1—C9—C5 | −1.4 (8) | C20—O3—C29—C30 | 177.7 (5) |
| C1—O1—C10—C11 | 179.5 (5) | O3—C29—C30—C31 | −64.3 (6) |
| O1—C10—C11—C12 | 63.8 (6) | C29—C30—C31—C32 | 177.2 (5) |
| C10—C11—C12—C13 | −173.9 (5) | N7—N6—C32—O4 | 176.0 (5) |
| N3—N2—C13—O2 | −176.0 (5) | N7—N6—C32—C31 | −5.4 (8) |
| N3—N2—C13—C12 | 4.5 (8) | C30—C31—C32—O4 | −0.4 (8) |
| C11—C12—C13—O2 | −1.8 (8) | C30—C31—C32—N6 | −179.0 (5) |
| C11—C12—C13—N2 | 177.7 (5) | N6—N7—C33—C34 | 180.0 (5) |
| N2—N3—C14—C15 | −178.6 (5) | N7—C33—C34—C38 | 175.0 (6) |
| N3—C14—C15—C19 | 3.4 (9) | N7—C33—C34—C35 | −5.0 (9) |
| N3—C14—C15—C16 | −177.0 (6) | C38—C34—C35—C36 | −0.5 (9) |
| C19—C15—C16—C17 | −0.2 (9) | C33—C34—C35—C36 | 179.5 (6) |
| C14—C15—C16—C17 | −179.8 (5) | C37—N8—C36—C35 | −0.9 (11) |
| C18—N4—C17—C16 | 1.3 (11) | C34—C35—C36—N8 | 0.5 (11) |
| C15—C16—C17—N4 | −1.2 (10) | C36—N8—C37—C38 | 1.4 (10) |
| C17—N4—C18—C19 | 0.0 (11) | N8—C37—C38—C34 | −1.5 (10) |
| C16—C15—C19—C18 | 1.4 (9) | C35—C34—C38—C37 | 0.9 (9) |
| C14—C15—C19—C18 | −179.1 (6) | C33—C34—C38—C37 | −179.1 (5) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C31—H31B···O3 | 0.97 | 2.55 | 2.908 (7) | 102 |
| C22—H22···O4$^{i}$ | 0.93 | 2.51 | 3.224 (7) | 134 |
| C12—H12B···O1 | 0.97 | 2.52 | 2.900 (7) | 103 |
| C7—H7···N8$^{ii}$ | 0.93 | 2.55 | 3.466 (9) | 167 |
| C3—H3···O2$^{iii}$ | 0.93 | 2.52 | 3.353 (7) | 150 |
| C2—H1···N4$^{iv}$ | 0.93 | 2.55 | 3.390 (9) | 150 |
| O5—H39···O4$^{v}$ | 0.85 | 2.17 | 2.964 (10) | 156 |
| N6—H6···N1$^{iii}$ | 0.86 | 2.10 | 2.934 (6) | 163 |
| N2—H2···N5$^{i}$ | 0.86 | 2.25 | 3.077 (7) | 161 |

Symmetry codes: (i) *x*, *y*, *z*−1; (ii) −*x*+3, *y*−1/2, −*z*+2; (iii) *x*, *y*, *z*+1; (iv) −*x*+1, *y*+1/2, −*z*+1; (v) −*x*+1, *y*+1/2, −*z*+2.

**Fig. 1**


O5
C27
C26
N5
N4
O4
C32
C30
O3
C28
C25
C18
C17
C20
C16
N6
C31
C29
C24
C19
C23
C15
N7
C21
C22
C14
C33
C4
C3
N3
C2
C12
C34
C9
N2
C38
C35
C5
C1
C13
C6
O1
C10
C11
C37
C36
N1
C7
O2
C8
N8