

7-Chloro-4-[(*E*)-2-(2,5-dimethoxybenzylidene)hydrazin-1-yl]quinoline

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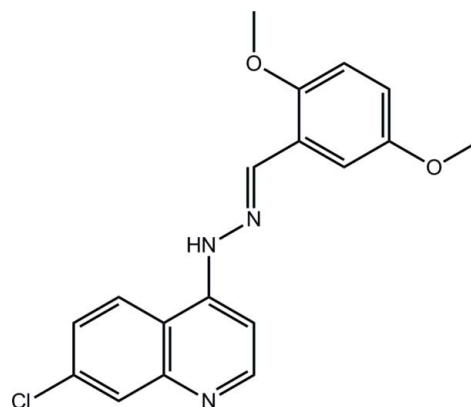
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.041; wR factor = 0.107; data-to-parameter ratio = 16.8.

In the nearly planar title compound (r.m.s. deviation for the 24 non-H atoms = 0.064 Å), $\text{C}_{18}\text{H}_{16}\text{ClN}_3\text{O}_2$, the conformation about the $\text{N}=\text{C}$ bond is *E*. Supramolecular chains propagated by glide symmetry along [001] are found in the crystal packing. These are sustained by $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds with the quinoline N atom being the acceptor. The chains are connected into a three-dimensional architecture by $\pi-\pi$ interactions involving all three aromatic rings [centroid-centroid distances = 3.5650 (9)–3.6264 (9) Å].

Related literature

For the biological activity, including anti-tubercular and anti-tumour activity, of compounds containing the quinolinyl nucleus, see: de Souza *et al.* (2009); Candea *et al.* (2009); Montenegro *et al.* (2011, 2012). For related structures, see: Howie *et al.* (2010); de Souza *et al.* (2010); de Lima Ferreira *et al.* (2010). For the synthesis, see: Montenegro *et al.* (2012).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{16}\text{ClN}_3\text{O}_2$
 $M_r = 341.79$
Monoclinic, $P2_1/c$
 $a = 10.5183$ (2) Å
 $b = 12.9132$ (3) Å
 $c = 12.9861$ (2) Å
 $\beta = 112.723$ (2)°

$V = 1626.93$ (5) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.25$ mm⁻¹
 $T = 120$ K
 $0.32 \times 0.20 \times 0.15$ mm

Data collection

Bruker–Nonius Roper CCD camera
on a κ -goniostat diffractometer
Absorption correction: multi-scan
(SADABS; Sheldrick, 2007)
 $T_{\min} = 0.652$, $T_{\max} = 0.746$

20405 measured reflections
3723 independent reflections
3067 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.107$
 $S = 1.03$
3723 reflections
222 parameters
1 restraint

H atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{\max} = 0.33$ e Å⁻³
 $\Delta\rho_{\min} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{N2}-\text{H2n}\cdots\text{N1}^i$ | 0.88 (1) | 2.19 (1) | 3.0572 (17) | 167 (2) |

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

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and DIAMOND (Brandenburg, 2006); software used to prepare material for publication: pubCIF (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5494).

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supplementary materials

Acta Cryst. (2012). E68, o1244–o1245 [doi:10.1107/S1600536812012871]

7-Chloro-4-[(*E*)-2-(2,5-dimethoxybenzylidene)hydrazin-1-yl]quinoline

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Comment

The quinoline nucleus is an important moiety found in various synthetic and natural products with a wide range of pharmacological activities (de Souza *et al.*, 2009), including anti-tubercular (Candea *et al.*, 2009) and anti-tumour (Montenegro *et al.*, 2012) activities. Among the derivatives studied have been arylaldehyde 7-chloroquinoline-4-hydrazones (Candea *et al.*, 2009; Montenegro *et al.*, 2011). Some crystal structures of these hydrazones, including related methoxy-substituted derivatives have been reported (Howie *et al.*, 2010; de Souza *et al.*, 2010; de Lima Ferreira *et al.*, 2010). We now wish to report the crystal structure of the title compound, (I).

In (I), Fig. 1, the entire molecule is planar with the r.m.s. deviation of all 24 non-hydrogen atoms being 0.064 Å. The maximum deviations from the least-squares plane are 0.108 (2) for the C5 atom and -0.165 (1) Å for the C11 atom. The conformation about the N3=C10 bond [1.2834 (18) Å] is *E*.

The most prominent feature of the crystal packing is the formation of supramolecular chains *via* N—H⋯N hydrogen bonds with the quinolinyl-N atom being the acceptor, Table 1. The chains are propagated by glide symmetry along the *c* axis, Fig. 2. Molecules are consolidated into a three-dimensional architecture by π — π interactions whereby the dimethoxybenzene ring interacts with both components of the quinolinyl residue along with symmetry related dimethoxybenzene rings [centroid(dimethoxybenzene)⋯centroid(NC₅)ⁱ; (C₆)ⁱ; (dimethoxybenzene)ⁱⁱ = 3.5650 (9), 3.6264 (9) and 3.5872 (9) Å, with angles of inclination = 2.36 (7) 4.20 (7) and 0° for symmetry operations *i*: 1 - *x*, -*y*, 1 - *z* and *ii*: 2 - *x*, -*y*, 1 - *z*]. The π — π interactions between the dimethoxybenzene and quinolinyl residues lead to zigzag layers in the *bc* plane and the π (dimethoxybenzene)⋯ π (dimethoxybenzene) interactions link these layers along the *a* axis, Fig. 3.

Experimental

The compound was prepared from 7-chloro-4-quinolinylhydrazone with 2,5-dimethoxybenzaldehyde (Montenegro *et al.*, 2012). The crystals used in the structure determination were grown from an ethanol solution of the compound.

Refinement

The C-bound H atoms were geometrically placed (C—H = 0.95–0.98 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2$ – $1.5U_{\text{eq}}(\text{C})$. The N-bound H-atom was located in a difference Fourier map and refined with a N—H distance = 0.88±0.01 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. Owing to poor agreement, the (1̄ 0 2) and (2 3 0) reflections were omitted from the final cycles of refinement.

Computing details

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular

graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

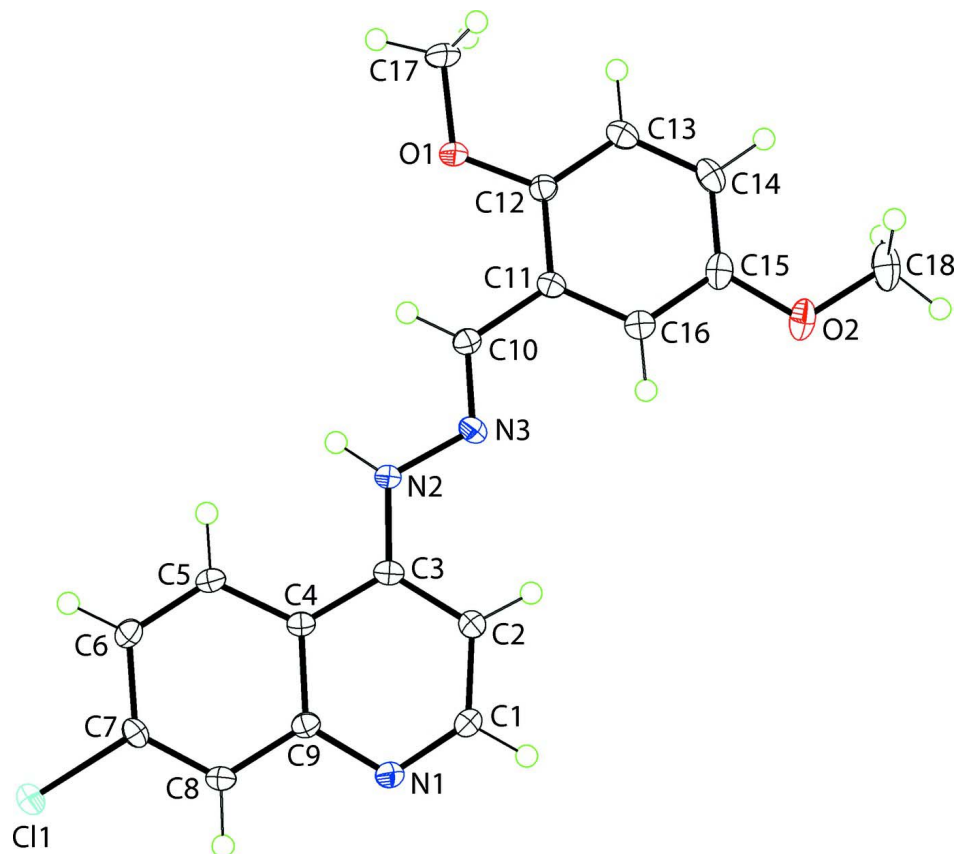


Figure 1

The molecular structure showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

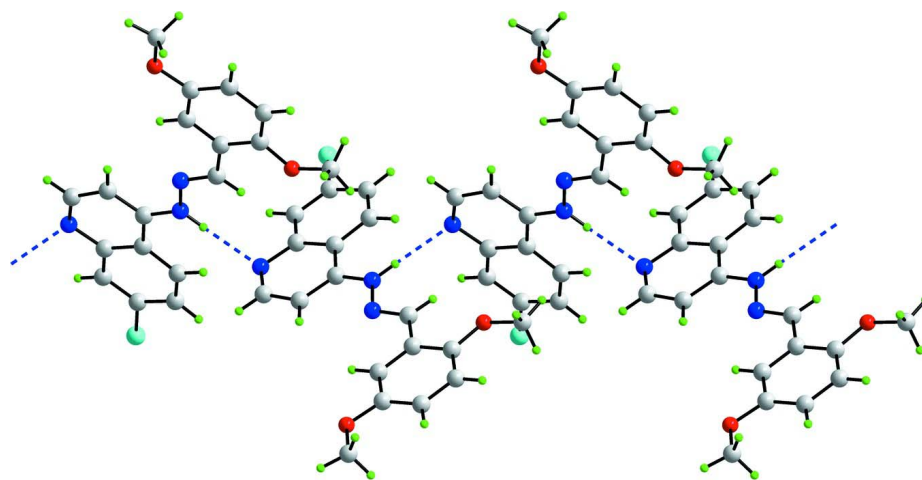


Figure 2

A view of the supramolecular chain along [001] in (I). The N—H...N hydrogen bonds are shown as blue dashed lines.

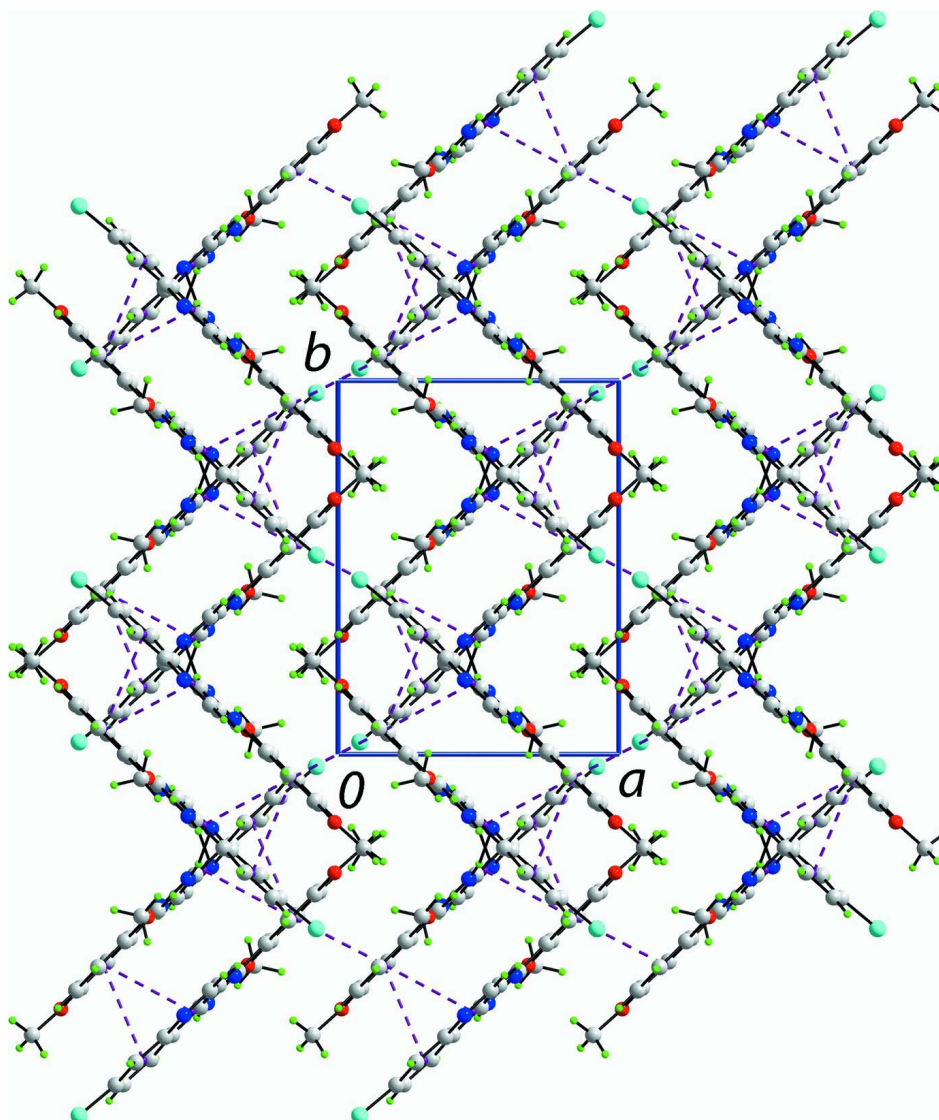


Figure 3

A view in projection down the *c* axis of unit-cell contents of (I). The N—H...N and $\pi\cdots\pi$ interactions are shown as blue and purple dashed lines, respectively.

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Crystal data

$C_{18}H_{16}ClN_3O_2$

$M_r = 341.79$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 10.5183(2)\ \text{\AA}$

$b = 12.9132(3)\ \text{\AA}$

$c = 12.9861(2)\ \text{\AA}$

$\beta = 112.723(2)^\circ$

$V = 1626.93(5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 712$

$D_x = 1.395\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 8419 reflections

$\theta = 2.9\text{--}27.5^\circ$

$\mu = 0.25\ \text{mm}^{-1}$

$T = 120\ \text{K}$

Prism, yellow

$0.32 \times 0.20 \times 0.15\ \text{mm}$

Data collection

Bruker–Nonius Roper CCD camera on a κ -goniostat
 diffractometer
 Radiation source: Bruker–Nonius FR591 rotating anode
 Graphite monochromator
 Detector resolution: 9.091 pixels mm⁻¹
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2007)

$T_{\min} = 0.652$, $T_{\max} = 0.746$
 20405 measured reflections
 3723 independent reflections
 3067 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$
 $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.2^\circ$
 $h = -13 \rightarrow 13$
 $k = -15 \rightarrow 16$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.107$
 $S = 1.03$
 3723 reflections
 222 parameters
 1 restraint
 Primary atom site location: structure-invariant direct methods

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
 $w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.6611P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| C11 | 0.07757 (4) | 0.46710 (3) | 0.65860 (3) | 0.02240 (13) |
| O1 | 0.67832 (11) | 0.06456 (8) | 0.25193 (8) | 0.0200 (2) |
| O2 | 0.98754 (12) | -0.18234 (9) | 0.60966 (10) | 0.0273 (3) |
| N1 | 0.45113 (13) | 0.19900 (10) | 0.83352 (10) | 0.0169 (3) |
| N2 | 0.53424 (13) | 0.16602 (10) | 0.54269 (10) | 0.0169 (3) |
| H2N | 0.4970 (16) | 0.2057 (11) | 0.4831 (10) | 0.020* |
| N3 | 0.63038 (13) | 0.09359 (10) | 0.54186 (10) | 0.0174 (3) |
| C1 | 0.54351 (15) | 0.13277 (12) | 0.82563 (12) | 0.0173 (3) |
| H1 | 0.5927 | 0.0921 | 0.8897 | 0.021* |
| C2 | 0.57480 (15) | 0.11751 (12) | 0.73189 (12) | 0.0172 (3) |
| H2 | 0.6407 | 0.0669 | 0.7327 | 0.021* |
| C3 | 0.50855 (15) | 0.17718 (11) | 0.63725 (12) | 0.0143 (3) |
| C4 | 0.40798 (14) | 0.25126 (11) | 0.64014 (11) | 0.0135 (3) |
| C5 | 0.33252 (15) | 0.31670 (11) | 0.54965 (12) | 0.0157 (3) |
| H5 | 0.3502 | 0.3143 | 0.4833 | 0.019* |

| | | | | |
|------|--------------|---------------|--------------|------------|
| C6 | 0.23425 (15) | 0.38355 (11) | 0.55596 (12) | 0.0168 (3) |
| H6 | 0.1850 | 0.4277 | 0.4950 | 0.020* |
| C7 | 0.20770 (15) | 0.38559 (11) | 0.65392 (12) | 0.0155 (3) |
| C8 | 0.27876 (15) | 0.32511 (11) | 0.74402 (12) | 0.0158 (3) |
| H8 | 0.2589 | 0.3287 | 0.8094 | 0.019* |
| C9 | 0.38202 (14) | 0.25713 (11) | 0.73985 (12) | 0.0142 (3) |
| C10 | 0.64283 (15) | 0.08142 (11) | 0.44812 (12) | 0.0157 (3) |
| H10 | 0.5869 | 0.1209 | 0.3851 | 0.019* |
| C11 | 0.74242 (15) | 0.00736 (11) | 0.43720 (12) | 0.0145 (3) |
| C12 | 0.75975 (15) | -0.00070 (11) | 0.33516 (12) | 0.0156 (3) |
| C13 | 0.85456 (16) | -0.07004 (12) | 0.32490 (13) | 0.0194 (3) |
| H13 | 0.8663 | -0.0752 | 0.2561 | 0.023* |
| C14 | 0.93308 (16) | -0.13246 (12) | 0.41476 (13) | 0.0203 (3) |
| H14 | 0.9977 | -0.1800 | 0.4070 | 0.024* |
| C15 | 0.91658 (15) | -0.12495 (12) | 0.51558 (13) | 0.0195 (3) |
| C16 | 0.82194 (15) | -0.05485 (11) | 0.52614 (13) | 0.0172 (3) |
| H16 | 0.8114 | -0.0494 | 0.5954 | 0.021* |
| C17 | 0.7013 (2) | 0.06604 (15) | 0.15086 (14) | 0.0303 (4) |
| H17A | 0.7981 | 0.0827 | 0.1674 | 0.045* |
| H17B | 0.6421 | 0.1185 | 0.1005 | 0.045* |
| H17C | 0.6796 | -0.0021 | 0.1151 | 0.045* |
| C18 | 1.09064 (17) | -0.25167 (13) | 0.60485 (16) | 0.0306 (4) |
| H18A | 1.0479 | -0.3035 | 0.5466 | 0.046* |
| H18B | 1.1349 | -0.2864 | 0.6771 | 0.046* |
| H18C | 1.1599 | -0.2127 | 0.5876 | 0.046* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|------------|------------|--------------|--------------|--------------|
| C11 | 0.0225 (2) | 0.0242 (2) | 0.0220 (2) | 0.00878 (15) | 0.01015 (16) | 0.00116 (15) |
| O1 | 0.0247 (6) | 0.0234 (6) | 0.0150 (5) | 0.0069 (5) | 0.0111 (5) | 0.0024 (4) |
| O2 | 0.0263 (6) | 0.0217 (6) | 0.0280 (6) | 0.0076 (5) | 0.0040 (5) | 0.0068 (5) |
| N1 | 0.0197 (6) | 0.0178 (6) | 0.0140 (6) | 0.0014 (5) | 0.0073 (5) | 0.0000 (5) |
| N2 | 0.0197 (7) | 0.0191 (7) | 0.0141 (6) | 0.0052 (5) | 0.0091 (5) | 0.0022 (5) |
| N3 | 0.0185 (6) | 0.0176 (6) | 0.0188 (6) | 0.0031 (5) | 0.0100 (5) | -0.0005 (5) |
| C1 | 0.0189 (8) | 0.0183 (7) | 0.0143 (7) | 0.0015 (6) | 0.0057 (6) | 0.0024 (6) |
| C2 | 0.0175 (7) | 0.0184 (7) | 0.0161 (7) | 0.0028 (6) | 0.0067 (6) | 0.0002 (6) |
| C3 | 0.0148 (7) | 0.0152 (7) | 0.0139 (7) | -0.0041 (5) | 0.0068 (6) | -0.0031 (5) |
| C4 | 0.0141 (7) | 0.0143 (7) | 0.0127 (7) | -0.0026 (5) | 0.0057 (6) | -0.0017 (5) |
| C5 | 0.0183 (7) | 0.0174 (7) | 0.0128 (7) | -0.0011 (6) | 0.0076 (6) | 0.0005 (6) |
| C6 | 0.0188 (7) | 0.0155 (7) | 0.0157 (7) | 0.0000 (6) | 0.0063 (6) | 0.0024 (6) |
| C7 | 0.0149 (7) | 0.0127 (7) | 0.0190 (7) | 0.0007 (5) | 0.0067 (6) | -0.0029 (6) |
| C8 | 0.0184 (7) | 0.0171 (7) | 0.0141 (7) | -0.0010 (6) | 0.0085 (6) | -0.0019 (6) |
| C9 | 0.0157 (7) | 0.0139 (7) | 0.0125 (7) | -0.0025 (6) | 0.0048 (5) | -0.0021 (5) |
| C10 | 0.0163 (7) | 0.0164 (7) | 0.0150 (7) | -0.0008 (6) | 0.0067 (6) | 0.0006 (6) |
| C11 | 0.0144 (7) | 0.0139 (7) | 0.0162 (7) | -0.0024 (5) | 0.0072 (6) | -0.0017 (5) |
| C12 | 0.0166 (7) | 0.0152 (7) | 0.0154 (7) | -0.0007 (6) | 0.0065 (6) | -0.0002 (5) |
| C13 | 0.0207 (8) | 0.0188 (8) | 0.0218 (8) | -0.0006 (6) | 0.0113 (6) | -0.0039 (6) |
| C14 | 0.0171 (7) | 0.0152 (7) | 0.0296 (8) | 0.0008 (6) | 0.0101 (7) | -0.0029 (6) |
| C15 | 0.0165 (7) | 0.0142 (7) | 0.0237 (8) | -0.0015 (6) | 0.0031 (6) | 0.0012 (6) |

| | | | | | | |
|-----|-------------|-------------|-------------|-------------|------------|-------------|
| C16 | 0.0179 (7) | 0.0162 (7) | 0.0176 (7) | -0.0029 (6) | 0.0071 (6) | -0.0014 (6) |
| C17 | 0.0427 (11) | 0.0363 (10) | 0.0193 (8) | 0.0131 (8) | 0.0202 (8) | 0.0059 (7) |
| C18 | 0.0236 (9) | 0.0184 (8) | 0.0431 (11) | 0.0048 (7) | 0.0053 (8) | 0.0037 (7) |

Geometric parameters (Å, °)

| | | | |
|------------|-------------|-------------|-------------|
| C11—C7 | 1.7462 (15) | C6—H6 | 0.9500 |
| O1—C12 | 1.3773 (18) | C7—C8 | 1.365 (2) |
| O1—C17 | 1.4235 (18) | C8—C9 | 1.414 (2) |
| O2—C15 | 1.3764 (18) | C8—H8 | 0.9500 |
| O2—C18 | 1.426 (2) | C10—C11 | 1.465 (2) |
| N1—C1 | 1.3277 (19) | C10—H10 | 0.9500 |
| N1—C9 | 1.3742 (18) | C11—C16 | 1.390 (2) |
| N2—C3 | 1.3631 (18) | C11—C12 | 1.409 (2) |
| N2—N3 | 1.3805 (17) | C12—C13 | 1.384 (2) |
| N2—H2N | 0.884 (9) | C13—C14 | 1.396 (2) |
| N3—C10 | 1.2834 (18) | C13—H13 | 0.9500 |
| C1—C2 | 1.392 (2) | C14—C15 | 1.389 (2) |
| C1—H1 | 0.9500 | C14—H14 | 0.9500 |
| C2—C3 | 1.389 (2) | C15—C16 | 1.391 (2) |
| C2—H2 | 0.9500 | C16—H16 | 0.9500 |
| C3—C4 | 1.437 (2) | C17—H17A | 0.9800 |
| C4—C5 | 1.416 (2) | C17—H17B | 0.9800 |
| C4—C9 | 1.4248 (19) | C17—H17C | 0.9800 |
| C5—C6 | 1.373 (2) | C18—H18A | 0.9800 |
| C5—H5 | 0.9500 | C18—H18B | 0.9800 |
| C6—C7 | 1.404 (2) | C18—H18C | 0.9800 |
| C12—O1—C17 | 117.14 (12) | N3—C10—C11 | 120.69 (13) |
| C15—O2—C18 | 117.38 (13) | N3—C10—H10 | 119.7 |
| C1—N1—C9 | 115.86 (12) | C11—C10—H10 | 119.7 |
| C3—N2—N3 | 118.64 (12) | C16—C11—C12 | 118.92 (13) |
| C3—N2—H2N | 123.5 (11) | C16—C11—C10 | 121.33 (13) |
| N3—N2—H2N | 117.7 (11) | C12—C11—C10 | 119.74 (13) |
| C10—N3—N2 | 115.71 (12) | O1—C12—C13 | 124.95 (13) |
| N1—C1—C2 | 125.86 (13) | O1—C12—C11 | 115.19 (13) |
| N1—C1—H1 | 117.1 | C13—C12—C11 | 119.85 (14) |
| C2—C1—H1 | 117.1 | C12—C13—C14 | 120.57 (14) |
| C3—C2—C1 | 119.21 (13) | C12—C13—H13 | 119.7 |
| C3—C2—H2 | 120.4 | C14—C13—H13 | 119.7 |
| C1—C2—H2 | 120.4 | C15—C14—C13 | 119.91 (14) |
| N2—C3—C2 | 122.16 (13) | C15—C14—H14 | 120.0 |
| N2—C3—C4 | 119.86 (13) | C13—C14—H14 | 120.0 |
| C2—C3—C4 | 117.96 (13) | O2—C15—C14 | 125.22 (14) |
| C5—C4—C9 | 118.67 (13) | O2—C15—C16 | 115.23 (14) |
| C5—C4—C3 | 123.90 (13) | C14—C15—C16 | 119.55 (14) |
| C9—C4—C3 | 117.42 (12) | C11—C16—C15 | 121.20 (14) |
| C6—C5—C4 | 121.28 (13) | C11—C16—H16 | 119.4 |
| C6—C5—H5 | 119.4 | C15—C16—H16 | 119.4 |
| C4—C5—H5 | 119.4 | O1—C17—H17A | 109.5 |

| | | | |
|--------------|--------------|-----------------|--------------|
| C5—C6—C7 | 118.92 (13) | O1—C17—H17B | 109.5 |
| C5—C6—H6 | 120.5 | H17A—C17—H17B | 109.5 |
| C7—C6—H6 | 120.5 | O1—C17—H17C | 109.5 |
| C8—C7—C6 | 122.10 (13) | H17A—C17—H17C | 109.5 |
| C8—C7—C11 | 119.49 (11) | H17B—C17—H17C | 109.5 |
| C6—C7—C11 | 118.41 (11) | O2—C18—H18A | 109.5 |
| C7—C8—C9 | 119.80 (13) | O2—C18—H18B | 109.5 |
| C7—C8—H8 | 120.1 | H18A—C18—H18B | 109.5 |
| C9—C8—H8 | 120.1 | O2—C18—H18C | 109.5 |
| N1—C9—C8 | 117.18 (12) | H18A—C18—H18C | 109.5 |
| N1—C9—C4 | 123.63 (13) | H18B—C18—H18C | 109.5 |
| C8—C9—C4 | 119.18 (13) | | |
| | | | |
| C3—N2—N3—C10 | 175.16 (13) | C3—C4—C9—N1 | 2.5 (2) |
| C9—N1—C1—C2 | -0.3 (2) | C5—C4—C9—C8 | 2.2 (2) |
| N1—C1—C2—C3 | 1.8 (2) | C3—C4—C9—C8 | -176.72 (13) |
| N3—N2—C3—C2 | -0.6 (2) | N2—N3—C10—C11 | 179.47 (12) |
| N3—N2—C3—C4 | -179.38 (12) | N3—C10—C11—C16 | 2.7 (2) |
| C1—C2—C3—N2 | -179.88 (14) | N3—C10—C11—C12 | -176.54 (14) |
| C1—C2—C3—C4 | -1.1 (2) | C17—O1—C12—C13 | -5.0 (2) |
| N2—C3—C4—C5 | -1.0 (2) | C17—O1—C12—C11 | 174.34 (14) |
| C2—C3—C4—C5 | -179.75 (14) | C16—C11—C12—O1 | -179.20 (13) |
| N2—C3—C4—C9 | 177.93 (13) | C10—C11—C12—O1 | 0.1 (2) |
| C2—C3—C4—C9 | -0.9 (2) | C16—C11—C12—C13 | 0.2 (2) |
| C9—C4—C5—C6 | -1.1 (2) | C10—C11—C12—C13 | 179.50 (13) |
| C3—C4—C5—C6 | 177.74 (13) | O1—C12—C13—C14 | 179.53 (14) |
| C4—C5—C6—C7 | -0.8 (2) | C11—C12—C13—C14 | 0.2 (2) |
| C5—C6—C7—C8 | 1.6 (2) | C12—C13—C14—C15 | -0.2 (2) |
| C5—C6—C7—C11 | -177.40 (11) | C18—O2—C15—C14 | 3.0 (2) |
| C6—C7—C8—C9 | -0.5 (2) | C18—O2—C15—C16 | -176.98 (13) |
| C11—C7—C8—C9 | 178.51 (11) | C13—C14—C15—O2 | 179.83 (14) |
| C1—N1—C9—C8 | 177.32 (13) | C13—C14—C15—C16 | -0.2 (2) |
| C1—N1—C9—C4 | -1.9 (2) | C12—C11—C16—C15 | -0.6 (2) |
| C7—C8—C9—N1 | 179.30 (13) | C10—C11—C16—C15 | -179.88 (13) |
| C7—C8—C9—C4 | -1.4 (2) | O2—C15—C16—C11 | -179.42 (13) |
| C5—C4—C9—N1 | -178.56 (13) | C14—C15—C16—C11 | 0.6 (2) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|--------------------------|-------------|---------------|-----------------------|-------------------------|
| N2—H2n...N1 ⁱ | 0.88 (1) | 2.19 (1) | 3.0572 (17) | 167 (2) |

Symmetry code: (i) *x*, -*y*+1/2, *z*-1/2.