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N'-(5-Chloro-2-hydroxybenzylidene)-nicotinohydrazide

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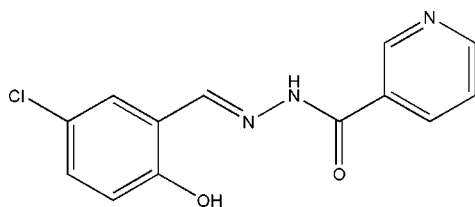
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
R factor = 0.049; wR factor = 0.115; data-to-parameter ratio = 15.2.

There are two independent Schiff base molecules in the asymmetric unit of the title compound, $\text{C}_{13}\text{H}_{10}\text{ClN}_3\text{O}_2$. The dihedral angles between the benzene and pyridine rings are 12.8 (2) and 1.9 (2)° in the two molecules. Intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds are observed. Molecules are linked into centrosymmetric $R_4^2(26)$ motifs by $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ interactions.

Related literature

For the biological properties of Schiff base compounds, see: Jeewoth *et al.* (1999); Ren *et al.* (2002); Eltayeb *et al.* (2008); Sinha *et al.* (2008). For metal complexes of Schiff base compounds, see: Shivakumar *et al.* (2008); Prabhakaran *et al.* (2006); Dhar *et al.* (2005). For related structures, see: Cui *et al.* (2007); Jing *et al.* (2007); Ma *et al.* (2008); Salhin *et al.* (2007); Lin *et al.* (2007); Alhadi *et al.* (2008); Xue *et al.* (2008); Wang *et al.* (2008); Lu (2008); Diao *et al.* (2008); Qiu (2009); Mohd Lair *et al.* (2009*a,b*). For reference structural data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{10}\text{ClN}_3\text{O}_2$
 $M_r = 275.69$
Monoclinic, $P2_1/n$
 $a = 9.792$ (2) Å
 $b = 23.358$ (3) Å
 $c = 10.926$ (2) Å
 $\beta = 96.848$ (2)°

$V = 2481.2$ (8) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.31$ mm⁻¹
 $T = 298$ K
 $0.30 \times 0.30 \times 0.27$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.913$, $T_{\max} = 0.921$
14387 measured reflections
5342 independent reflections
3193 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.115$
 $S = 1.01$
5342 reflections
351 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{O1}-\text{H1}\cdots\text{N1}$ | 0.82 | 1.88 | 2.594 (2) | 145 |
| $\text{O3}-\text{H3}\cdots\text{N4}$ | 0.82 | 1.82 | 2.538 (2) | 146 |
| $\text{N5}-\text{H5}\cdots\text{N3}^i$ | 0.891 (10) | 2.109 (11) | 2.991 (3) | 171 (2) |
| $\text{N2}-\text{H2}\cdots\text{O3}$ | 0.892 (10) | 2.097 (10) | 2.984 (2) | 173 (2) |

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2215).

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supplementary materials

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N'-(5-Chloro-2-hydroxybenzylidene)nicotinohydrazide

C.-G. Ren

Comment

The Schiff base compounds show excellent biological properties (Jeewoth *et al.*, 1999; Ren *et al.*, 2002; Eltayeb *et al.*, 2008; Sinha *et al.*, 2008). Moreover, the Schiff base compounds have been widely used as versatile ligands in coordination chemistry (Shivakumar *et al.*, 2008; Prabhakaran *et al.*, 2006; Dhar *et al.*, 2005). We report here the crystal structure of the title compound. In the title compound, Fig. 1, there are two independent molecules in the asymmetric unit. The dihedral angles between the benzene and pyridine rings are 12.8 (2) and 1.9 (2)°, respectively. All the bond lengths are within normal values (Allen *et al.*, 1987) and comparable to those in other similar compounds (Cui *et al.*, 2007; Jing *et al.*, 2007; Ma *et al.*, 2008; Salhin *et al.*, 2007; Lin *et al.*, 2007; Alhadi *et al.*, 2008; Xue *et al.*, 2008; Wang *et al.*, 2008; Lu, 2008; Diao *et al.*, 2008; Qiu, 2009; Lair *et al.*, 2009a,b). The molecules of the title compound are linked into centrosymmetric $R^4_4(26)$ motifs by N–H···O and N–H···N interactions (Table 1, Fig. 2) (Bernstein *et al.*, 1995).

Experimental

All the starting materials were obtained with AR grade from Lancaster. 5-Chloro-2-hydroxybenzaldehyde (1.0 mmol, 157.1 mg) and nicotinohydrazide (1.0 mmol, 137.1 mg) were refluxed in a 30 ml methanol solution for 30 min to give a clear yellow solution. Yellow block-shaped single crystals of the compound were obtained by slow evaporation of the solution for five days at room temperature.

Refinement

H2 and H5 were located from a difference Fourier map and refined isotropically, with the N–H distance restrained to 0.90 (1) Å, and with U_{iso} restrained to 0.08 Å². Other H atoms were constrained to ideal geometries, with $d(\text{C–H}) = 0.93$ Å, $d(\text{O–H}) = 0.82$ Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{O})$.

Figures

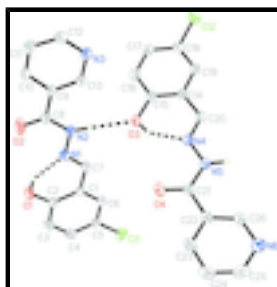


Fig. 1. The molecular structure of the compound with 30% probability ellipsoids. The intramolecular O–H···N hydrogen bonds are shown as dashed lines.

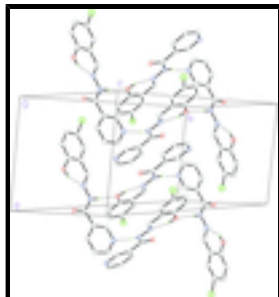


Fig. 2. Molecular packing of the compound with hydrogen bonds drawn as dashed lines. Hydrogen atoms not involved in hydrogen bonding have been omitted.

(I)

Crystal data

$C_{13}H_{10}ClN_3O_2$

$M_r = 275.69$

Monoclinic, $P2_1/n$

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

$a = 9.792\ (2)\ \text{\AA}$

$b = 23.358\ (3)\ \text{\AA}$

$c = 10.926\ (2)\ \text{\AA}$

$\beta = 96.848\ (2)^\circ$

$V = 2481.2\ (8)\ \text{\AA}^3$

$Z = 8$

$F_{000} = 1136$

$D_x = 1.476\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2236 reflections

$\theta = 2.6\text{--}24.5^\circ$

$\mu = 0.31\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Block, yellow

$0.30 \times 0.30 \times 0.27\ \text{mm}$

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298\ \text{K}$

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.913$, $T_{\max} = 0.921$

14387 measured reflections

5342 independent reflections

3193 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\max} = 27.0^\circ$

$\theta_{\min} = 1.7^\circ$

$h = -12 \rightarrow 12$

$k = -29 \rightarrow 27$

$l = -12 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

$wR(F^2) = 0.115$

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

$$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.01$ $(\Delta/\sigma)_{\max} < 0.001$
 5342 reflections $\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$
 351 parameters $\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$
 2 restraints Extinction correction: none
 Primary atom site location: structure-invariant direct methods

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|---------------|--------------|----------------------------------|
| C11 | -0.31666 (7) | 0.15824 (3) | 0.49570 (7) | 0.0648 (2) |
| C12 | 0.67723 (7) | -0.10226 (3) | 0.57393 (6) | 0.0648 (2) |
| N1 | 0.28860 (19) | 0.22587 (8) | 0.70899 (16) | 0.0414 (5) |
| N2 | 0.4128 (2) | 0.20261 (8) | 0.75424 (18) | 0.0421 (5) |
| N3 | 0.7948 (2) | 0.13722 (8) | 0.92250 (17) | 0.0438 (5) |
| N4 | 0.29718 (18) | 0.00154 (8) | 0.94498 (16) | 0.0387 (4) |
| N5 | 0.2156 (2) | -0.01091 (8) | 1.03441 (16) | 0.0402 (5) |
| N6 | -0.0434 (2) | -0.04219 (10) | 1.3111 (2) | 0.0659 (6) |
| O1 | 0.13564 (17) | 0.31528 (6) | 0.65672 (16) | 0.0520 (4) |
| H1 | 0.2075 | 0.2981 | 0.6784 | 0.078* |
| O2 | 0.50725 (19) | 0.29034 (7) | 0.76057 (19) | 0.0755 (6) |
| O3 | 0.39503 (17) | 0.07872 (6) | 0.81707 (15) | 0.0486 (4) |
| H3 | 0.3484 | 0.0658 | 0.8682 | 0.073* |
| O4 | 0.17570 (18) | 0.08396 (7) | 1.05029 (15) | 0.0598 (5) |
| C1 | 0.0545 (2) | 0.21766 (9) | 0.63088 (19) | 0.0368 (5) |
| C2 | 0.0344 (2) | 0.27712 (9) | 0.62187 (19) | 0.0394 (5) |
| C3 | -0.0937 (3) | 0.29868 (10) | 0.5765 (2) | 0.0496 (6) |
| H3A | -0.1072 | 0.3381 | 0.5715 | 0.059* |
| C4 | -0.2008 (3) | 0.26227 (11) | 0.5390 (2) | 0.0504 (6) |
| H4 | -0.2865 | 0.2770 | 0.5088 | 0.060* |
| C5 | -0.1810 (2) | 0.20380 (10) | 0.5461 (2) | 0.0438 (6) |
| C6 | -0.0555 (2) | 0.18151 (10) | 0.59222 (19) | 0.0415 (6) |
| H6 | -0.0439 | 0.1420 | 0.5977 | 0.050* |
| C7 | 0.1862 (2) | 0.19285 (10) | 0.6792 (2) | 0.0411 (6) |
| H7 | 0.1957 | 0.1534 | 0.6882 | 0.049* |
| C8 | 0.5195 (2) | 0.23961 (10) | 0.7816 (2) | 0.0444 (6) |

supplementary materials

| | | | | |
|-----|-------------|---------------|--------------|------------|
| C9 | 0.6530 (2) | 0.21501 (9) | 0.83757 (19) | 0.0359 (5) |
| C10 | 0.7653 (3) | 0.25086 (10) | 0.8548 (2) | 0.0483 (6) |
| H10 | 0.7567 | 0.2890 | 0.8304 | 0.058* |
| C11 | 0.8900 (3) | 0.23037 (11) | 0.9080 (2) | 0.0585 (7) |
| H11 | 0.9658 | 0.2545 | 0.9224 | 0.070* |
| C12 | 0.9001 (3) | 0.17355 (11) | 0.9395 (2) | 0.0509 (6) |
| H12 | 0.9849 | 0.1597 | 0.9746 | 0.061* |
| C13 | 0.6739 (2) | 0.15881 (9) | 0.8743 (2) | 0.0425 (6) |
| H13 | 0.5985 | 0.1343 | 0.8649 | 0.051* |
| C14 | 0.4402 (2) | -0.02227 (9) | 0.79497 (18) | 0.0357 (5) |
| C15 | 0.4588 (2) | 0.03497 (9) | 0.7643 (2) | 0.0380 (5) |
| C16 | 0.5456 (2) | 0.04896 (10) | 0.6773 (2) | 0.0476 (6) |
| H16 | 0.5579 | 0.0872 | 0.6572 | 0.057* |
| C17 | 0.6134 (2) | 0.00709 (10) | 0.6205 (2) | 0.0464 (6) |
| H17 | 0.6724 | 0.0169 | 0.5632 | 0.056* |
| C18 | 0.5936 (2) | -0.04957 (10) | 0.6489 (2) | 0.0446 (6) |
| C19 | 0.5080 (2) | -0.06441 (9) | 0.7344 (2) | 0.0414 (6) |
| H19 | 0.4951 | -0.1028 | 0.7523 | 0.050* |
| C20 | 0.3537 (2) | -0.03840 (9) | 0.88893 (19) | 0.0395 (5) |
| H20 | 0.3400 | -0.0767 | 0.9076 | 0.047* |
| C21 | 0.1567 (2) | 0.03509 (10) | 1.0846 (2) | 0.0419 (6) |
| C22 | 0.0660 (2) | 0.02299 (9) | 1.18211 (19) | 0.0401 (5) |
| C23 | 0.0067 (3) | 0.06862 (11) | 1.2362 (2) | 0.0538 (7) |
| H23 | 0.0236 | 0.1059 | 1.2124 | 0.065* |
| C24 | -0.0774 (3) | 0.05834 (13) | 1.3256 (2) | 0.0651 (8) |
| H24 | -0.1193 | 0.0885 | 1.3624 | 0.078* |
| C25 | -0.0986 (3) | 0.00294 (14) | 1.3595 (2) | 0.0657 (8) |
| H25 | -0.1554 | -0.0034 | 1.4206 | 0.079* |
| C26 | 0.0382 (3) | -0.03068 (10) | 1.2238 (2) | 0.0542 (7) |
| H26 | 0.0790 | -0.0616 | 1.1887 | 0.065* |
| H5 | 0.206 (3) | -0.0477 (5) | 1.053 (2) | 0.080* |
| H2 | 0.411 (3) | 0.1663 (5) | 0.779 (2) | 0.080* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|--------------|-------------|
| C11 | 0.0444 (4) | 0.0649 (5) | 0.0827 (5) | -0.0128 (3) | -0.0025 (4) | 0.0037 (4) |
| C12 | 0.0697 (5) | 0.0567 (4) | 0.0709 (5) | 0.0172 (4) | 0.0197 (4) | -0.0037 (3) |
| N1 | 0.0369 (12) | 0.0387 (11) | 0.0479 (11) | 0.0041 (9) | 0.0020 (9) | 0.0047 (9) |
| N2 | 0.0351 (12) | 0.0342 (11) | 0.0552 (12) | 0.0012 (9) | -0.0016 (10) | 0.0047 (9) |
| N3 | 0.0362 (12) | 0.0371 (11) | 0.0574 (13) | 0.0001 (9) | 0.0023 (10) | 0.0029 (9) |
| N4 | 0.0374 (12) | 0.0354 (11) | 0.0432 (11) | -0.0026 (9) | 0.0048 (9) | 0.0047 (9) |
| N5 | 0.0422 (12) | 0.0333 (11) | 0.0465 (11) | 0.0001 (9) | 0.0116 (10) | 0.0040 (9) |
| N6 | 0.0726 (17) | 0.0628 (15) | 0.0674 (15) | 0.0088 (13) | 0.0299 (13) | 0.0101 (12) |
| O1 | 0.0514 (11) | 0.0317 (9) | 0.0697 (11) | -0.0002 (8) | -0.0060 (10) | 0.0021 (8) |
| O2 | 0.0604 (13) | 0.0346 (11) | 0.1240 (16) | -0.0032 (9) | -0.0194 (12) | 0.0196 (10) |
| O3 | 0.0549 (11) | 0.0292 (9) | 0.0649 (11) | -0.0015 (8) | 0.0206 (9) | 0.0034 (7) |
| O4 | 0.0713 (13) | 0.0321 (10) | 0.0788 (12) | 0.0011 (9) | 0.0205 (10) | 0.0045 (9) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0369 (14) | 0.0353 (13) | 0.0390 (13) | 0.0020 (10) | 0.0073 (10) | 0.0041 (10) |
| C2 | 0.0432 (15) | 0.0366 (13) | 0.0387 (13) | -0.0007 (11) | 0.0057 (11) | 0.0002 (10) |
| C3 | 0.0506 (17) | 0.0368 (14) | 0.0600 (16) | 0.0088 (12) | 0.0013 (13) | 0.0051 (11) |
| C4 | 0.0419 (16) | 0.0554 (17) | 0.0527 (16) | 0.0080 (13) | 0.0008 (12) | 0.0060 (12) |
| C5 | 0.0358 (15) | 0.0476 (15) | 0.0481 (14) | -0.0051 (11) | 0.0056 (11) | 0.0018 (11) |
| C6 | 0.0430 (15) | 0.0354 (13) | 0.0467 (14) | -0.0024 (11) | 0.0079 (11) | 0.0033 (10) |
| C7 | 0.0426 (15) | 0.0326 (13) | 0.0482 (14) | 0.0022 (11) | 0.0052 (12) | 0.0025 (10) |
| C8 | 0.0445 (16) | 0.0353 (14) | 0.0523 (15) | 0.0002 (11) | 0.0012 (12) | 0.0038 (11) |
| C9 | 0.0376 (14) | 0.0289 (12) | 0.0410 (12) | -0.0028 (10) | 0.0042 (10) | -0.0003 (9) |
| C10 | 0.0498 (17) | 0.0308 (13) | 0.0635 (16) | -0.0067 (11) | 0.0035 (13) | 0.0037 (11) |
| C11 | 0.0433 (16) | 0.0477 (16) | 0.0815 (19) | -0.0131 (13) | -0.0043 (14) | 0.0003 (14) |
| C12 | 0.0367 (15) | 0.0520 (17) | 0.0625 (16) | 0.0016 (12) | 0.0000 (12) | 0.0000 (12) |
| C13 | 0.0338 (14) | 0.0358 (13) | 0.0571 (15) | -0.0048 (11) | 0.0027 (12) | 0.0019 (11) |
| C14 | 0.0332 (13) | 0.0330 (12) | 0.0399 (13) | -0.0017 (10) | -0.0006 (10) | 0.0050 (10) |
| C15 | 0.0335 (13) | 0.0355 (13) | 0.0444 (13) | -0.0027 (10) | 0.0020 (11) | 0.0005 (10) |
| C16 | 0.0530 (16) | 0.0365 (14) | 0.0542 (15) | -0.0072 (12) | 0.0097 (13) | 0.0051 (11) |
| C17 | 0.0454 (16) | 0.0496 (15) | 0.0457 (14) | -0.0044 (12) | 0.0121 (12) | 0.0040 (12) |
| C18 | 0.0449 (15) | 0.0427 (14) | 0.0455 (14) | 0.0065 (11) | 0.0029 (12) | 0.0007 (11) |
| C19 | 0.0445 (15) | 0.0330 (13) | 0.0459 (13) | 0.0020 (11) | 0.0025 (11) | 0.0062 (10) |
| C20 | 0.0417 (14) | 0.0281 (12) | 0.0482 (14) | -0.0016 (10) | 0.0037 (11) | 0.0049 (10) |
| C21 | 0.0393 (14) | 0.0365 (14) | 0.0484 (14) | -0.0008 (11) | -0.0004 (11) | -0.0002 (11) |
| C22 | 0.0367 (14) | 0.0385 (14) | 0.0439 (13) | 0.0042 (10) | 0.0005 (11) | -0.0027 (10) |
| C23 | 0.0542 (17) | 0.0453 (15) | 0.0622 (17) | 0.0041 (13) | 0.0073 (14) | -0.0108 (12) |
| C24 | 0.0600 (19) | 0.068 (2) | 0.0687 (19) | 0.0135 (15) | 0.0139 (15) | -0.0213 (15) |
| C25 | 0.0514 (18) | 0.094 (2) | 0.0538 (17) | 0.0100 (17) | 0.0166 (14) | -0.0020 (16) |
| C26 | 0.0606 (18) | 0.0439 (16) | 0.0619 (16) | 0.0083 (13) | 0.0230 (14) | -0.0006 (12) |

Geometric parameters (Å, °)

| | | | |
|---------|------------|---------|-----------|
| C11—C5 | 1.740 (2) | C7—H7 | 0.9300 |
| C12—C18 | 1.736 (2) | C8—C9 | 1.491 (3) |
| N1—C7 | 1.276 (3) | C9—C10 | 1.376 (3) |
| N1—N2 | 1.369 (2) | C9—C13 | 1.381 (3) |
| N2—C8 | 1.361 (3) | C10—C11 | 1.375 (3) |
| N2—H2 | 0.892 (10) | C10—H10 | 0.9300 |
| N3—C12 | 1.332 (3) | C11—C12 | 1.372 (3) |
| N3—C13 | 1.336 (3) | C11—H11 | 0.9300 |
| N4—C20 | 1.278 (3) | C12—H12 | 0.9300 |
| N4—N5 | 1.365 (2) | C13—H13 | 0.9300 |
| N5—C21 | 1.365 (3) | C14—C15 | 1.396 (3) |
| N5—H5 | 0.891 (10) | C14—C19 | 1.397 (3) |
| N6—C25 | 1.324 (3) | C14—C20 | 1.456 (3) |
| N6—C26 | 1.342 (3) | C15—C16 | 1.388 (3) |
| O1—C2 | 1.354 (3) | C16—C17 | 1.371 (3) |
| O1—H1 | 0.8200 | C16—H16 | 0.9300 |
| O2—C8 | 1.210 (2) | C17—C18 | 1.378 (3) |
| O3—C15 | 1.361 (2) | C17—H17 | 0.9300 |
| O3—H3 | 0.8200 | C18—C19 | 1.373 (3) |
| O4—C21 | 1.223 (2) | C19—H19 | 0.9300 |

supplementary materials

| | | | |
|------------|-------------|-------------|-------------|
| C1—C6 | 1.394 (3) | C20—H20 | 0.9300 |
| C1—C2 | 1.405 (3) | C21—C22 | 1.493 (3) |
| C1—C7 | 1.455 (3) | C22—C26 | 1.372 (3) |
| C2—C3 | 1.387 (3) | C22—C23 | 1.380 (3) |
| C3—C4 | 1.375 (3) | C23—C24 | 1.372 (3) |
| C3—H3A | 0.9300 | C23—H23 | 0.9300 |
| C4—C5 | 1.380 (3) | C24—C25 | 1.369 (4) |
| C4—H4 | 0.9300 | C24—H24 | 0.9300 |
| C5—C6 | 1.374 (3) | C25—H25 | 0.9300 |
| C6—H6 | 0.9300 | C26—H26 | 0.9300 |
| C7—N1—N2 | 119.29 (19) | N3—C12—C11 | 123.3 (2) |
| C8—N2—N1 | 116.92 (19) | N3—C12—H12 | 118.3 |
| C8—N2—H2 | 125.2 (17) | C11—C12—H12 | 118.3 |
| N1—N2—H2 | 116.2 (18) | N3—C13—C9 | 124.5 (2) |
| C12—N3—C13 | 116.7 (2) | N3—C13—H13 | 117.7 |
| C20—N4—N5 | 120.75 (18) | C9—C13—H13 | 117.7 |
| C21—N5—N4 | 115.52 (18) | C15—C14—C19 | 118.6 (2) |
| C21—N5—H5 | 127.1 (17) | C15—C14—C20 | 121.4 (2) |
| N4—N5—H5 | 117.4 (17) | C19—C14—C20 | 120.05 (19) |
| C25—N6—C26 | 115.5 (2) | O3—C15—C16 | 117.57 (19) |
| C2—O1—H1 | 109.5 | O3—C15—C14 | 122.55 (19) |
| C15—O3—H3 | 109.5 | C16—C15—C14 | 119.9 (2) |
| C6—C1—C2 | 118.8 (2) | C17—C16—C15 | 120.7 (2) |
| C6—C1—C7 | 119.2 (2) | C17—C16—H16 | 119.6 |
| C2—C1—C7 | 122.0 (2) | C15—C16—H16 | 119.6 |
| O1—C2—C3 | 117.5 (2) | C16—C17—C18 | 119.7 (2) |
| O1—C2—C1 | 122.7 (2) | C16—C17—H17 | 120.2 |
| C3—C2—C1 | 119.8 (2) | C18—C17—H17 | 120.2 |
| C4—C3—C2 | 120.5 (2) | C19—C18—C17 | 120.6 (2) |
| C4—C3—H3A | 119.8 | C19—C18—C12 | 120.18 (18) |
| C2—C3—H3A | 119.8 | C17—C18—C12 | 119.23 (18) |
| C3—C4—C5 | 119.9 (2) | C18—C19—C14 | 120.5 (2) |
| C3—C4—H4 | 120.1 | C18—C19—H19 | 119.7 |
| C5—C4—H4 | 120.1 | C14—C19—H19 | 119.7 |
| C6—C5—C4 | 120.6 (2) | N4—C20—C14 | 118.06 (19) |
| C6—C5—C11 | 120.01 (19) | N4—C20—H20 | 121.0 |
| C4—C5—C11 | 119.37 (19) | C14—C20—H20 | 121.0 |
| C5—C6—C1 | 120.4 (2) | O4—C21—N5 | 121.7 (2) |
| C5—C6—H6 | 119.8 | O4—C21—C22 | 121.3 (2) |
| C1—C6—H6 | 119.8 | N5—C21—C22 | 116.94 (19) |
| N1—C7—C1 | 119.2 (2) | C26—C22—C23 | 116.9 (2) |
| N1—C7—H7 | 120.4 | C26—C22—C21 | 124.6 (2) |
| C1—C7—H7 | 120.4 | C23—C22—C21 | 118.4 (2) |
| O2—C8—N2 | 121.6 (2) | C24—C23—C22 | 119.3 (2) |
| O2—C8—C9 | 121.2 (2) | C24—C23—H23 | 120.4 |
| N2—C8—C9 | 117.3 (2) | C22—C23—H23 | 120.4 |
| C10—C9—C13 | 116.8 (2) | C25—C24—C23 | 118.8 (2) |
| C10—C9—C8 | 117.9 (2) | C25—C24—H24 | 120.6 |
| C13—C9—C8 | 125.3 (2) | C23—C24—H24 | 120.6 |

| | | | |
|----------------|--------------|-----------------|--------------|
| C11—C10—C9 | 120.0 (2) | N6—C25—C24 | 124.2 (3) |
| C11—C10—H10 | 120.0 | N6—C25—H25 | 117.9 |
| C9—C10—H10 | 120.0 | C24—C25—H25 | 117.9 |
| C12—C11—C10 | 118.5 (2) | N6—C26—C22 | 125.3 (2) |
| C12—C11—H11 | 120.8 | N6—C26—H26 | 117.4 |
| C10—C11—H11 | 120.8 | C22—C26—H26 | 117.4 |
| C7—N1—N2—C8 | 178.3 (2) | C8—C9—C13—N3 | 178.8 (2) |
| C20—N4—N5—C21 | -177.7 (2) | C19—C14—C15—O3 | 178.9 (2) |
| C6—C1—C2—O1 | -179.7 (2) | C20—C14—C15—O3 | -2.0 (3) |
| C7—C1—C2—O1 | 0.3 (3) | C19—C14—C15—C16 | -1.3 (3) |
| C6—C1—C2—C3 | 0.8 (3) | C20—C14—C15—C16 | 177.9 (2) |
| C7—C1—C2—C3 | -179.2 (2) | O3—C15—C16—C17 | 179.9 (2) |
| O1—C2—C3—C4 | 179.7 (2) | C14—C15—C16—C17 | 0.1 (3) |
| C1—C2—C3—C4 | -0.7 (3) | C15—C16—C17—C18 | 1.0 (4) |
| C2—C3—C4—C5 | -0.2 (4) | C16—C17—C18—C19 | -0.8 (4) |
| C3—C4—C5—C6 | 1.0 (3) | C16—C17—C18—C12 | 178.62 (18) |
| C3—C4—C5—C11 | -178.88 (18) | C17—C18—C19—C14 | -0.5 (3) |
| C4—C5—C6—C1 | -1.0 (3) | C12—C18—C19—C14 | -179.87 (17) |
| C11—C5—C6—C1 | 178.92 (16) | C15—C14—C19—C18 | 1.5 (3) |
| C2—C1—C6—C5 | 0.1 (3) | C20—C14—C19—C18 | -177.7 (2) |
| C7—C1—C6—C5 | -179.9 (2) | N5—N4—C20—C14 | -179.66 (18) |
| N2—N1—C7—C1 | -179.88 (18) | C15—C14—C20—N4 | -1.5 (3) |
| C6—C1—C7—N1 | 176.7 (2) | C19—C14—C20—N4 | 177.7 (2) |
| C2—C1—C7—N1 | -3.3 (3) | N4—N5—C21—O4 | 0.5 (3) |
| N1—N2—C8—O2 | -3.9 (3) | N4—N5—C21—C22 | 179.61 (18) |
| N1—N2—C8—C9 | 176.60 (18) | O4—C21—C22—C26 | 178.9 (2) |
| O2—C8—C9—C10 | -5.6 (3) | N5—C21—C22—C26 | -0.2 (3) |
| N2—C8—C9—C10 | 173.9 (2) | O4—C21—C22—C23 | -2.0 (3) |
| O2—C8—C9—C13 | 174.2 (2) | N5—C21—C22—C23 | 178.9 (2) |
| N2—C8—C9—C13 | -6.3 (3) | C26—C22—C23—C24 | -1.1 (4) |
| C13—C9—C10—C11 | -0.9 (3) | C21—C22—C23—C24 | 179.7 (2) |
| C8—C9—C10—C11 | 178.9 (2) | C22—C23—C24—C25 | 0.8 (4) |
| C9—C10—C11—C12 | 2.0 (4) | C26—N6—C25—C24 | 0.2 (4) |
| C13—N3—C12—C11 | -1.3 (4) | C23—C24—C25—N6 | -0.4 (4) |
| C10—C11—C12—N3 | -0.8 (4) | C25—N6—C26—C22 | -0.6 (4) |
| C12—N3—C13—C9 | 2.5 (3) | C23—C22—C26—N6 | 1.1 (4) |
| C10—C9—C13—N3 | -1.3 (3) | C21—C22—C26—N6 | -179.8 (2) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|--------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O1—H1 \cdots N1 | 0.82 | 1.88 | 2.594 (2) | 145 |
| O3—H3 \cdots N4 | 0.82 | 1.82 | 2.538 (2) | 146 |
| N5—H5 \cdots N3 ⁱ | 0.891 (10) | 2.109 (11) | 2.991 (3) | 171 (2) |
| N2—H2 \cdots O3 | 0.892 (10) | 2.097 (10) | 2.984 (2) | 173 (2) |

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

Fig. 1

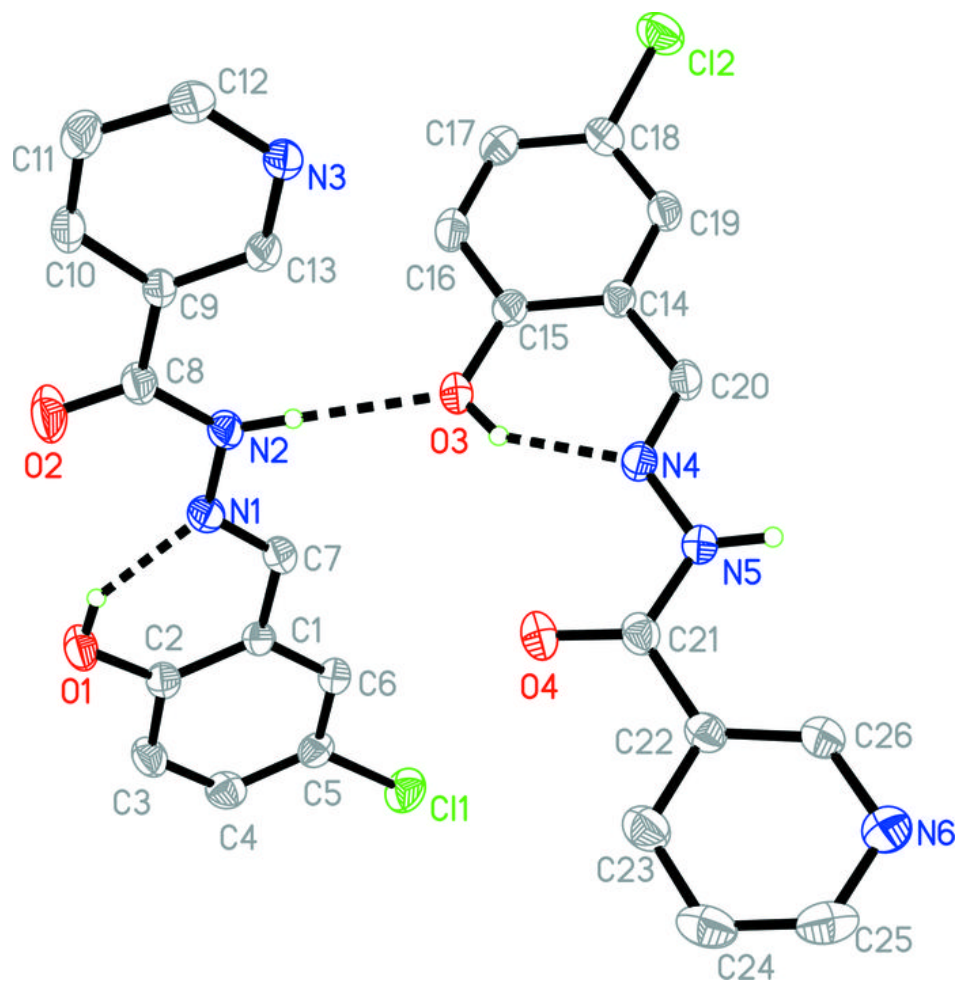


Fig. 2

