

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 3-(2-Pyridyl)-N'-salicylidenepropionohydrazide

Arne Roth, Axel Buchholz, Martin Gärtner, Helmar Görls and Winfried Plass\*

Institut für Anorganische und Analytische Chemie, Friedrich-Schiller-Universität Jena, Carl-Zeiss-Promenade 10, 07745 Jena, Germany  
Correspondence e-mail: sekr.plass@uni-jena.de

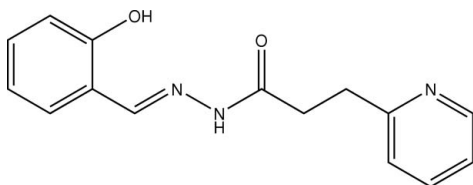
Received 4 May 2007; accepted 25 May 2007

Key indicators: single-crystal X-ray study;  $T = 183$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.035;  $wR$  factor = 0.087; data-to-parameter ratio = 9.9.

The Schiff base-type title compound,  $\text{C}_{15}\text{H}_{15}\text{N}_3\text{O}_2$ , was obtained by the reaction of 3-(2-pyridyl)propionohydrazide with salicylaldehyde in ethanol. Whereas the  $^1\text{H}$  NMR spectrum in solution points to a mixture of two isomers, only one isomer was found in the solid state. The phenolic OH group forms an intramolecular hydrogen bond with the imino N atom. Intermolecular hydrogen bonds between the amido NH function and the pyridyl N atom join the molecules into chains parallel to the  $a$  axis. The compound is achiral, but crystallizes in the space group  $P2_12_12_1$ , with the molecule adopting a chiral conformation.

## Related literature

For related literature on  $N$ -salicylidenehydrazides and their metal complexes, see: Johnson *et al.* (1982); Mohan *et al.* (1987*a,b*); Koh *et al.* (1998); Ainscough *et al.* (1999); Nica *et al.* (2005); Pohlmann *et al.* (2005); Becher *et al.* (2006); Roth *et al.* (2007). For synthetic procedures, see: Walter *et al.* (1941); Doering *et al.* (1947); Hallinan *et al.* (1993).



## Experimental

## Crystal data

 $\text{C}_{15}\text{H}_{15}\text{N}_3\text{O}_2$   
 $M_r = 269.30$ Orthorhombic,  $P2_12_12_1$   
 $a = 5.6638$  (2) Å  
 $b = 15.1845$  (5) Å  
 $c = 15.5994$  (4) Å $V = 1341.58$  (7) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 0.09$  mm<sup>-1</sup> $T = 183$  (2) K $0.9 \times 0.8 \times 0.4$  mm

## Data collection

Nonius KappaCCD area-detector  
diffractometer  
Absorption correction: none  
9539 measured reflections1793 independent reflections  
1592 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.040$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.035$  $wR(F^2) = 0.087$  $S = 1.06$ 

1793 reflections

182 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.15$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.17$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.84	1.85	2.585 (2)	145
$\text{N2}-\text{H2}\cdots\text{N3}^i$	0.88	2.15	2.952 (2)	152

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

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

The authors thank the Deutsche Forschungsgemeinschaft (SFB 436 'Metal-Mediated Reactions Modelled after Nature') for financial support. AR gratefully acknowledges support by a Graduiertenstipendium of the Freistaat Thüringen.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2074).

## References

- Ainscough, E. W., Brodie, A. M., Denny, W. A., Finlay, G. J., Gothe, S. A. & Ranford, J. D. (1999). *J. Inorg. Biochem.* **77**, 125–133.
- Becher, J., Seidel, I., Plass, W. & Klemm, D. (2006). *Tetrahedron*, **62**, 5675–5681.
- Doering, W. E. & Weil, R. A. N. (1947). *J. Am. Chem. Soc.* **69**, 2461–2466.
- Hallinan, E. A., Hagen, T. J., Husa, R. K., Tsymbalov, S., Rao, S. N., van Hoek, J.-P., Rafferty, M. F., Stapelfeld, A., Savage, M. A. & Reichman, M. (1993). *J. Med. Chem.* **36**, 3293–3299.
- Johnson, D. K., Murphy, T. B., Rose, N. J., Goodwin, W. H. & Pickart, L. (1982). *Inorg. Chim. Acta*, **67**, 159–165.
- Koh, L. L., Kon, O. L., Loh, K. W., Long, Y. C., Ranford, J. D., Tan, A. L. C. & Tjan, Y. Y. (1998). *J. Inorg. Biochem.* **72**, 155–162.
- Mohan, M., Gupta, N. S., Kumar, A. & Kumar, M. (1987*a*). *Inorg. Chim. Acta*, **135**, 167–177.
- Mohan, M., Kumar, A., Kumar, M. & Jha, N. K. (1987*b*). *Inorg. Chim. Acta*, **136**, 65–74.
- Nica, S., Pohlmann, A. & Plass, W. (2005). *Eur. J. Inorg. Chem.* pp. 2032–2036.
- Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Pohlmann, A., Nica, S., Luong, T. K. K. & Plass, W. (2005). *Inorg. Chem. Commun.* **8**, 289–292.
- Roth, A., Buchholz, A. & Plass, W. (2007). *Z. Anorg. Allg. Chem.* **633**, 383–392.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Siemens (1990). *XP*. Version 4.2. Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.
- Walter, L. A., Hunt, W. H. & Fosbinder, R. J. (1941). *J. Am. Chem. Soc.* **63**, 2771–2773.

**supplementary materials**

*Acta Cryst.* (2007). E63, o3073 [ doi:10.1107/S1600536807025664 ]

### 3-(2-Pyridyl)-*N'*-salicylidenepropionohydrazide

A. Roth, A. Buchholz, M. Gärtner, H. Görls and W. Plass

#### Comment

*N*-Salicylidenehydrazides and their metal complexes show a wide range of biological activities (Johnson *et al.*, 1982; Mohan *et al.*, 1987a; Koh *et al.*, 1998; Mohan *et al.*, 1987b; Ainscough *et al.*, 1999}. Most of the *N*-salicylidenehydrazide ligands reported so far provide hydrophobic alkylic or aryl side chains. However, in the past few years, new Schiff-base ligands bearing hydroxy and amino functions in the side chains have been synthesized to enable assembly of molecules in the crystal *via* hydrogen-bond interactions (Nica *et al.*, 2005; Pohlmann *et al.*, 2005; Becher *et al.*, 2006; Roth *et al.*, 2007). In this paper we report the crystal structure of the new Schiff-base ligand *N*-salicylidene-3-(2-pyridyl)-propionic acid hydrazide (H<sub>2</sub>salhypyp).

The compound was obtained by Schiff-base reaction of 3-(2-pyridyl)-propionic acid hydrazide with salicylaldehyde in ethanol. The NMR spectra revealed a splitting of most signals, indicating the presence of two isomers in solution, probably caused by *cis/trans* isomerization of the amide function. According to the <sup>1</sup>H NMR spectra (DMSO, 300 K), the ratio of isomers is 1.8:1. The crystal structure of H<sub>2</sub>salhypyp revealed only one isomer, with *trans* configuration of the amide group (Fig. 1). The phenolic O–H group forms an intramolecular hydrogen bond to the imino nitrogen atom N1. Intermolecular hydrogen bonds between the amido N–H function and the pyridyl nitrogen N3 join the molecules into chains parallel to the *a* axis. H<sub>2</sub>salhypyp, which is achiral, crystallizes in the orthorhombic space group *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, with the molecules adopting a chiral conformation.

#### Experimental

IR spectra were measured on a Bruker IFS55/Equinox spectrometer. Mass spectra were carried out on a MATSSQ-710 Bruker instrument. Elemental analyses were acquired by use of a LECO CHN/932 elemental analyzer. NMR spectra were recorded on a Bruker Avance 400 MHz spectrometer.

3-(2-Pyridyl)-propionic acid hydrazide was prepared according to described methods, *i.e.* reaction of potassium cyanide with 2-(2-pyridyl)-ethylbromide (Walter *et al.*, 1941), hydrolysis of 3-(2-pyridyl)-propionitrile with hydrochloric acid (Doering *et al.*, 1947), acid catalyzed esterification of 3-(2-pyridyl)-propionic acid in dry ethanol, and hydrazinolysis of the corresponding ethylester (Hallinan *et al.*, 1993).

3-(2-Pyridyl)-propionic acid hydrazide (650 mg, 3.94 mmol) dissolved in 10 ml ethanol was reacted at RT with one equivalent of salicylaldehyde and stirred for 1 h. The title compound (920 mg, 3.43 mmol) was obtained as microcrystalline colourless solid after leaving the reaction solution at 277 K over night. Recrystallization from ethanol (slow evaporation) lead to single crystals suitable for X-ray crystallography. The NMR spectra indicate the existence of two isomers in solution. C<sub>15</sub>H<sub>15</sub>N<sub>3</sub>O<sub>2</sub> (269.3): calcd. C 66.90, H 5.6, N 15.60, found C 66.95, H 5.59, N 15.92. IR (KBr): 3156 (ν(N–H)), 1684 (Amide I and ν(C=N)) cm<sup>-1</sup>. <sup>1</sup>H-NMR (DMSO, 400 MHz): 2.68 (t, <sup>3</sup>J=7.50 Hz, CH<sub>2</sub>–CO, major isomer); 3.06 (m, CH<sub>2</sub>–py, both isomers, and CH<sub>2</sub>–CO, minor isomer); 6.86 (2H, m, HC<sub>Ph</sub>); 7.24 (3H, m, 2 HC<sub>py</sub> and 1 HC<sub>Ph</sub>); 7.47 and 7.58 (1H, m, HC<sub>Ph</sub>);

## supplementary materials

7.68 (1H, m, HC<sub>py</sub>); 8.26 and 8.33 (1H, s, HC=N); 8.47 (1H, m, HC<sub>py</sub>); 10.12 and 11.17 (1H, s, NH); 11.24 and 11.66 (1H, s, OH) p.p.m.. <sup>13</sup>C-NMR (DMSO, 50 MHz): 31.3, 31.9, 32.4, and 32.9 (CH<sub>2</sub>-py and CH<sub>2</sub>-CO, both isomers); 116.1 and 116.3 (C<sub>Ph</sub>), 118.6 and 119.9 (C<sub>Ph</sub>), 119.2 and 119.4 (C<sub>Ph</sub>), 121.2 and 121.3 (C<sub>py</sub>), 122.8 (C<sub>Ph</sub>), 126.8 and 129.4 (C<sub>Ph</sub>), 130.8 and 131.1 (C<sub>Ph</sub>), 136.3 and 136.4 (C<sub>py</sub>), 141.0 and 146.4 (C=N), 148.9 (C<sub>py</sub>), 156.3 and 157.3 (C-OH), 160.0 and 160.5 (C<sub>py</sub>), 167.8 and 173.1 (C=O) p.p.m.. MS (EI): *m/z* = 269 (*M*<sup>+</sup>), 134 (100%, [py-CH<sub>2</sub>-CH<sub>2</sub>-CO]<sup>+</sup>), 106 ([py-CH<sub>2</sub>-CH<sub>2</sub>]<sup>+</sup>).

### Refinement

H atoms were positioned geometrically, *C*(*sp*<sup>2</sup>)-H = 0.95 Å, *C*(methylene)-H = 0.99 Å, O-H = 0.84 Å and N-H = 0.88 Å, and treated as riding atoms with displacement parameters, *U*<sub>iso</sub>(H) = *xU*<sub>eq</sub>(C), where *x* = 1.5 for O-H and 1.2 for all others. In the absence of significant anomalous scattering effects, Friedel pairs were averaged prior to the final refinement and the absolute structure was not determined.

### Figures

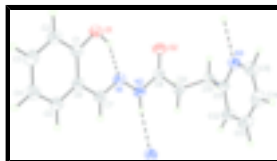


Fig. 1. Molecular structure of H<sub>2</sub>salpyph. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown with dashed line.

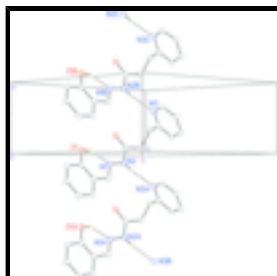


Fig. 2. Chains of H<sub>2</sub>salpyph molecules formed *via* N—H...N hydrogen bonding. Hydrogen bonds are shown with dashed lines. Only H atoms from the N—H and O—H groups are shown.

### *N*'-(2-Hydroxybenzylidene)-3-(2-pyridyl)propionohydrazide

#### Crystal data

C<sub>15</sub>H<sub>15</sub>N<sub>3</sub>O<sub>2</sub>

*M<sub>r</sub>* = 269.30

Orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>

Hall symbol: *P* 2ac 2ab

*a* = 5.6638 (2) Å

*b* = 15.1845 (5) Å

*c* = 15.5994 (4) Å

*V* = 1341.58 (7) Å<sup>3</sup>

*Z* = 4

*F*<sub>000</sub> = 568

*D<sub>x</sub>* = 1.333 Mg m<sup>-3</sup>

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 1793 reflections

θ = 2.6–27.5°

μ = 0.09 mm<sup>-1</sup>

*T* = 183 (2) K

Prism, colourless

0.9 × 0.8 × 0.4 mm

*Data collection*

Nonius KappaCCD area-detector diffractometer	1592 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.040$
Monochromator: graphite	$\theta_{\text{max}} = 27.5^\circ$
$T = 183(2)$ K	$\theta_{\text{min}} = 2.6^\circ$
$\varphi$ and $\omega$ scans	$h = -7 \rightarrow 6$
Absorption correction: none	$k = -18 \rightarrow 19$
9539 measured reflections	$l = -20 \rightarrow 20$
1793 independent reflections	

*Refinement*

Refinement on $F^2$	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.2563P]$
$R[F^2 > 2\sigma(F^2)] = 0.035$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.087$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.15 \text{ e } \text{\AA}^{-3}$
1793 reflections	$\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$
182 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

*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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.0597 (2)	0.55099 (9)	0.02570 (8)	0.0313 (3)
H1	0.0539	0.5574	0.0792	0.047*
O2	0.2491 (3)	0.54059 (11)	0.25857 (9)	0.0450 (4)
N1	-0.1275 (3)	0.59566 (9)	0.17046 (9)	0.0271 (3)

## supplementary materials

---

N2	-0.1174 (3)	0.60063 (10)	0.25852 (9)	0.0289 (4)
H2	-0.2369	0.6212	0.2885	0.035*
N3	0.5681 (3)	0.72701 (10)	0.34594 (9)	0.0297 (4)
C1	-0.1289 (3)	0.59146 (11)	-0.01074 (11)	0.0260 (4)
C2	-0.1355 (4)	0.59712 (12)	-0.10001 (12)	0.0311 (4)
H2A	-0.0104	0.5733	-0.1333	0.037*
C3	-0.3244 (4)	0.63740 (13)	-0.13968 (12)	0.0353 (5)
H3	-0.3287	0.6405	-0.2005	0.042*
C4	-0.5084 (4)	0.67347 (13)	-0.09247 (13)	0.0345 (5)
H4	-0.6372	0.7012	-0.1207	0.041*
C5	-0.5022 (4)	0.66865 (12)	-0.00396 (12)	0.0306 (4)
H5	-0.6273	0.6936	0.0285	0.037*
C6	-0.3142 (3)	0.62750 (11)	0.03849 (11)	0.0256 (4)
C7	-0.3087 (3)	0.62669 (12)	0.13202 (11)	0.0279 (4)
H7	-0.4383	0.6490	0.1640	0.033*
C8	0.0833 (4)	0.57268 (12)	0.29746 (11)	0.0283 (4)
C9	0.0853 (4)	0.58529 (12)	0.39373 (11)	0.0288 (4)
H9A	0.0566	0.5280	0.4222	0.035*
H9B	-0.0435	0.6259	0.4103	0.035*
C10	0.3216 (3)	0.62271 (12)	0.42386 (11)	0.0284 (4)
H10A	0.3242	0.6225	0.4873	0.034*
H10B	0.4496	0.5834	0.4038	0.034*
C11	0.3724 (3)	0.71506 (11)	0.39298 (10)	0.0240 (4)
C12	0.2289 (4)	0.78513 (12)	0.41561 (12)	0.0322 (4)
H12	0.0887	0.7750	0.4475	0.039*
C13	0.2894 (4)	0.86972 (13)	0.39182 (13)	0.0389 (5)
H13	0.1929	0.9182	0.4078	0.047*
C14	0.4915 (4)	0.88287 (13)	0.34455 (13)	0.0381 (5)
H14	0.5389	0.9403	0.3276	0.046*
C15	0.6227 (4)	0.80995 (13)	0.32266 (12)	0.0362 (5)
H15	0.7604	0.8187	0.2890	0.043*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0280 (7)	0.0345 (7)	0.0314 (6)	0.0050 (6)	0.0022 (6)	-0.0032 (5)
O2	0.0366 (9)	0.0611 (10)	0.0375 (7)	0.0196 (8)	0.0029 (7)	-0.0125 (7)
N1	0.0303 (9)	0.0258 (7)	0.0251 (7)	0.0002 (7)	0.0025 (7)	-0.0022 (6)
N2	0.0299 (9)	0.0330 (8)	0.0237 (7)	0.0046 (8)	0.0035 (7)	-0.0039 (6)
N3	0.0266 (8)	0.0333 (8)	0.0293 (7)	0.0028 (7)	0.0031 (7)	-0.0002 (6)
C1	0.0260 (9)	0.0208 (8)	0.0311 (9)	-0.0023 (8)	0.0006 (8)	-0.0001 (6)
C2	0.0322 (11)	0.0305 (9)	0.0307 (9)	-0.0016 (9)	0.0049 (8)	-0.0025 (7)
C3	0.0425 (12)	0.0348 (10)	0.0285 (9)	-0.0014 (10)	-0.0009 (9)	0.0011 (7)
C4	0.0353 (11)	0.0320 (9)	0.0363 (10)	0.0011 (9)	-0.0050 (9)	0.0032 (8)
C5	0.0278 (10)	0.0285 (9)	0.0354 (10)	0.0002 (8)	0.0014 (8)	-0.0018 (7)
C6	0.0270 (10)	0.0205 (8)	0.0293 (9)	-0.0024 (8)	0.0012 (8)	-0.0010 (6)
C7	0.0293 (10)	0.0238 (8)	0.0306 (9)	-0.0011 (8)	0.0037 (8)	-0.0017 (7)
C8	0.0283 (10)	0.0261 (8)	0.0304 (9)	0.0016 (8)	0.0006 (8)	-0.0038 (7)

C9	0.0307 (10)	0.0278 (9)	0.0277 (9)	-0.0014 (8)	0.0017 (8)	0.0006 (7)
C10	0.0303 (10)	0.0258 (9)	0.0292 (9)	0.0015 (8)	-0.0030 (8)	0.0031 (7)
C11	0.0249 (9)	0.0268 (8)	0.0204 (7)	0.0007 (8)	-0.0016 (7)	-0.0002 (6)
C12	0.0332 (10)	0.0312 (9)	0.0324 (9)	0.0037 (9)	0.0111 (9)	-0.0003 (8)
C13	0.0494 (13)	0.0265 (9)	0.0408 (11)	0.0067 (10)	0.0058 (10)	-0.0007 (8)
C14	0.0485 (13)	0.0298 (10)	0.0358 (10)	-0.0072 (10)	-0.0002 (10)	0.0050 (8)
C15	0.0334 (11)	0.0443 (11)	0.0309 (9)	-0.0055 (10)	0.0063 (9)	0.0050 (8)

*Geometric parameters (Å, °)*

O1—C1	1.357 (2)	C5—H5	0.9500
O1—H1	0.8400	C6—C7	1.459 (2)
O2—C8	1.219 (2)	C7—H7	0.9500
N1—C7	1.279 (2)	C8—C9	1.514 (2)
N1—N2	1.377 (2)	C9—C10	1.528 (3)
N2—C8	1.357 (3)	C9—H9A	0.9900
N2—H2	0.8800	C9—H9B	0.9900
N3—C11	1.342 (2)	C10—C11	1.510 (2)
N3—C15	1.347 (3)	C10—H10A	0.9900
C1—C2	1.396 (2)	C10—H10B	0.9900
C1—C6	1.411 (3)	C11—C12	1.385 (3)
C2—C3	1.379 (3)	C12—C13	1.380 (3)
C2—H2A	0.9500	C12—H12	0.9500
C3—C4	1.389 (3)	C13—C14	1.376 (3)
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.383 (3)	C14—C15	1.376 (3)
C4—H4	0.9500	C14—H14	0.9500
C5—C6	1.401 (3)	C15—H15	0.9500
C1—O1—H1	109.5	N2—C8—C9	114.24 (17)
C7—N1—N2	118.75 (16)	C8—C9—C10	111.02 (16)
C8—N2—N1	117.65 (16)	C8—C9—H9A	109.4
C8—N2—H2	121.2	C10—C9—H9A	109.4
N1—N2—H2	121.2	C8—C9—H9B	109.4
C11—N3—C15	117.62 (17)	C10—C9—H9B	109.4
O1—C1—C2	117.85 (17)	H9A—C9—H9B	108.0
O1—C1—C6	122.21 (15)	C11—C10—C9	114.47 (15)
C2—C1—C6	119.94 (18)	C11—C10—H10A	108.6
C3—C2—C1	119.73 (19)	C9—C10—H10A	108.6
C3—C2—H2A	120.1	C11—C10—H10B	108.6
C1—C2—H2A	120.1	C9—C10—H10B	108.6
C2—C3—C4	121.29 (17)	H10A—C10—H10B	107.6
C2—C3—H3	119.4	N3—C11—C12	121.37 (17)
C4—C3—H3	119.4	N3—C11—C10	117.23 (16)
C5—C4—C3	119.29 (19)	C12—C11—C10	121.34 (17)
C5—C4—H4	120.4	C13—C12—C11	120.05 (18)
C3—C4—H4	120.4	C13—C12—H12	120.0
C4—C5—C6	120.99 (19)	C11—C12—H12	120.0
C4—C5—H5	119.5	C14—C13—C12	119.05 (19)
C6—C5—H5	119.5	C14—C13—H13	120.5

## supplementary materials

---

C5—C6—C1	118.76 (16)	C12—C13—H13	120.5
C5—C6—C7	119.51 (17)	C13—C14—C15	117.72 (19)
C1—C6—C7	121.65 (17)	C13—C14—H14	121.1
N1—C7—C6	119.29 (17)	C15—C14—H14	121.1
N1—C7—H7	120.4	N3—C15—C14	124.16 (19)
C6—C7—H7	120.4	N3—C15—H15	117.9
O2—C8—N2	123.18 (16)	C14—C15—H15	117.9
O2—C8—C9	122.58 (18)		

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 $\cdots$ N1	0.84	1.85	2.585 (2)	145
N2—H2 $\cdots$ N3 <sup>i</sup>	0.88	2.15	2.952 (2)	152

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



Fig. 1

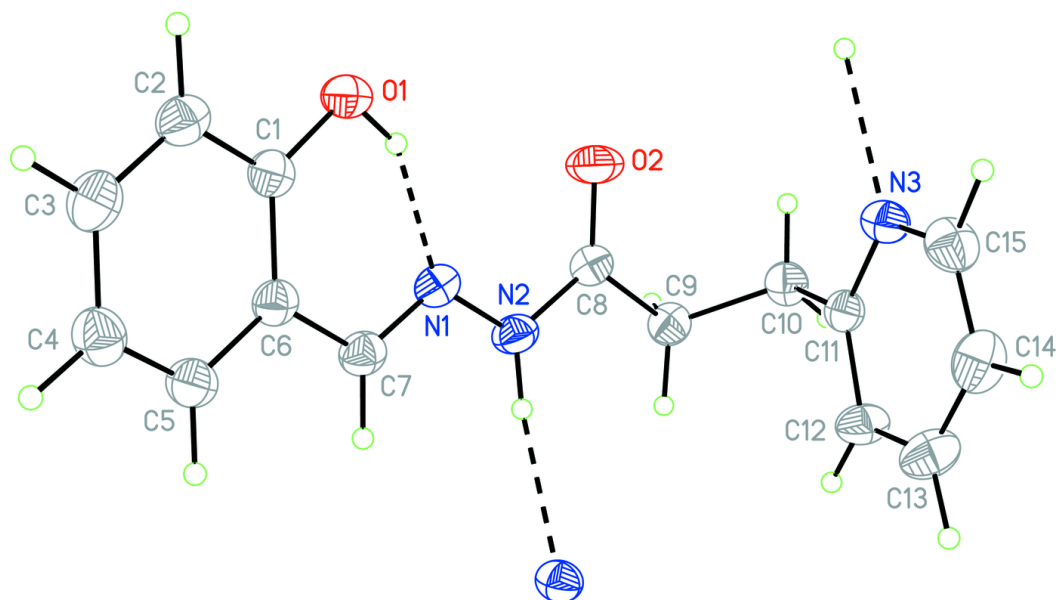


Fig. 2

