

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-Aminopyridin-3-ol

Richard Betz,* Thomas Gerber, Eric Hosten and Henk Schalekamp

Nelson Mandela Metropolitan University, Summerstrand Campus, Department of Chemistry, University Way, Summerstrand, PO Box 77000, Port Elizabeth 6031, South Africa

Correspondence e-mail: richard.betz@webmail.co.za

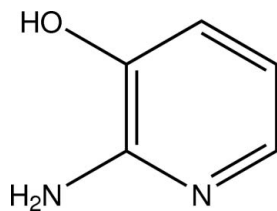
Received 3 August 2011; accepted 24 August 2011

Key indicators: single-crystal X-ray study; $T = 200$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.036; wR factor = 0.117; data-to-parameter ratio = 15.9.

The molecule of the title pyridine derivative, $\text{C}_5\text{H}_6\text{N}_2\text{O}$, shows approximate C_s symmetry. Intracyclic angles cover the range 118.34 (10)– 123.11 (10)°. In the crystal, $\text{O}-\text{H}\cdots\text{N}$, $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds connect the molecules into double layers perpendicular to the a axis. The shortest centroid-centroid distance between two π -systems is 3.8887 (7) Å.

Related literature

For the crystal structure of 2,3-diaminopyridine, see: Betz *et al.* (2011). For graph-set analysis of hydrogen bonds, see: Etter *et al.* (1990); Bernstein *et al.* (1995). For general information about the chelate effect in coordination chemistry, see: Gade (1998).



Experimental

Crystal data

$\text{C}_5\text{H}_6\text{N}_2\text{O}$
 $M_r = 110.12$
 Monoclinic, $P2_1/c$

$a = 12.5310$ (6) Å
 $b = 3.8887$ (2) Å
 $c = 11.6042$ (5) Å

$\beta = 113.139$ (2)°
 $V = 519.98$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.10$ mm⁻¹
 $T = 200$ K
 $0.29 \times 0.25 \times 0.13$ mm

Data collection

Bruker APEXII CCD
 diffractometer
 4820 measured reflections

1289 independent reflections
 1008 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.117$
 $S = 1.13$
 1289 reflections
 81 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{O1}-\text{H1}\cdots\text{N1}^{\text{i}}$ | 0.82 | 1.85 | 2.6639 (12) | 172 |
| $\text{N2}-\text{H71}\cdots\text{O1}^{\text{ii}}$ | 0.870 (17) | 2.276 (17) | 3.0184 (13) | 143.2 (12) |
| $\text{N2}-\text{H72}\cdots\text{N2}^{\text{iii}}$ | 0.895 (18) | 2.358 (17) | 3.1249 (15) | 143.8 (15) |

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

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT*; 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 *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

The authors thank Mrs Valerie Jacobs for helpful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2372).

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
 Betz, R., Gerber, T., Hosten, E. & Schalekamp, H. (2011). *Acta Cryst.* **E67**, o2154.
 Bruker (2010). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Gade, L. H. (1998). *Koordinationschemie*, 1. Auflage. Weinheim: Wiley-VCH.
 Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supplementary materials

Acta Cryst. (2011). E67, o2513 [doi:10.1107/S1600536811034775]

2-Aminopyridin-3-ol

R. Betz, T. Gerber, E. Hosten and H. Schalekamp

Comment

Chelate ligands have found widespread use in coordination chemistry due to the enhanced thermodynamic stability of resultant metal complexes in relation to coordination compounds exclusively applying comparable monodentate ligands (Gade, 1998). Combining different donor atoms, a molecular set-up to accommodate a large variety of metal centers of variable Lewis acidity is at hand. In this aspect, the title compound seemed interesting due to its use as strictly neutral or – depending on the pH value – as anionic or cationic ligand. Furthermore, thanks to the presence of three possible donor atoms, the title compound might serve as a building block in the formation of metal-organic framework structures. At the beginning of a more comprehensive study to elucidate the formation of coordination polymers featuring mixed *N,O* ligands, we determined the structure of the title compound to enable comparative studies of metrical parameters in envisioned reaction products. Information about the molecular and crystal structure of 2,3-diaminopyridine is apparent in the literature (Betz *et al.*, 2011).

Intracyclic angles range from 118.34 (10) ° to 123.11 (10) ° with the smallest angle found on the carbon atom bearing the hydroxyl group and the largest angle found on the unsubstituted carbon atom in *ortho* position to the intracyclic N atom. The molecule is essentially planar (r.m.s. of all fitted non-hydrogen atoms = 0.0092 Å). The amino group is not planar, the least-squares planes defined by the atoms of the heterocycle on the one hand and the atoms of the NH₂ group on the other hand group enclose an angle of 30.73(1.69) ° (Fig. 1).

The crystal structure of the title compound is marked by hydrogen bonds (Fig. 2). While the hydroxyl group forms a hydrogen bond to the intracyclic N atom (and its O atom acts as acceptor for one of the NH₂ supported hydrogen bonds), there is also a cooperative hydrogen bonding system of the NH₂⋯NH₂-type. In terms of graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995), the descriptor for these interactions on the unitary level is C¹₁(2)C¹₁(5)C¹₁(5). In total, the molecules are connected to double layers perpendicular to the crystallographic *a* axis. The shortest intercentroid distance between two π-systems was measured at 3.8887 (7) Å.

The packing of the title compound in the crystal is shown in Figure 3.

Experimental

The compound was obtained commercially (Aldrich). Crystals suitable for the X-ray diffraction study were taken directly from the provided compound.

Refinement

Carbon-bound H atoms were placed in calculated positions (C—H 0.95 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2*U*_{eq}(C). The H atom of the hydroxyl group was placed in a calculated position

supplementary materials

(O—H 0.82 Å) and was included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.5U_{\text{eq}}(\text{O})$. Both nitrogen-bound H atoms were located on a difference Fourier map and refined freely.

Figures

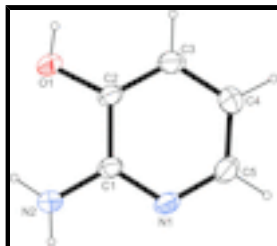


Fig. 1. The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level).

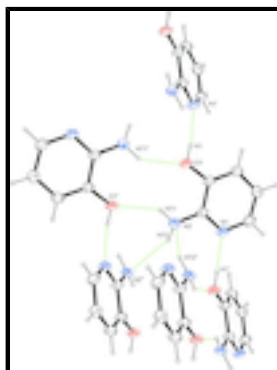


Fig. 2. Intermolecular contacts, viewed approximately along $[-1 -1 -1]$. Symmetry operators: ⁱ $x, -y + 3/2, z + 1/2$; ⁱⁱ $-x + 1, -y + 1, -z + 1$; ⁱⁱⁱ $-x + 1, y - 1/2, -z + 1/2$; ^{vi} $-x + 1, y + 1/2, -z + 1/2$; ^v $x, -y + 3/2, z - 1/2$.

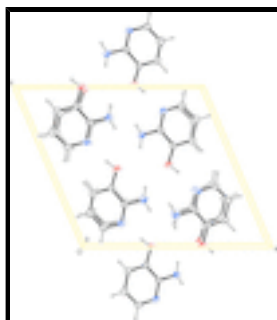


Fig. 3. Molecular packing of the title compound, viewed along $[0 1 0]$ (anisotropic displacement ellipsoids drawn at 50% probability level).

2-Aminopyridin-3-ol

Crystal data

$\text{C}_5\text{H}_6\text{N}_2\text{O}$

$M_r = 110.12$

Monoclinic, $P2_1/c$

Hall symbol: $-P 2ybc$

$a = 12.5310 (6) \text{ \AA}$

$b = 3.8887 (2) \text{ \AA}$

$c = 11.6042 (5) \text{ \AA}$

$\beta = 113.139 (2)^\circ$

$V = 519.98 (4) \text{ \AA}^3$

$F(000) = 232$

$D_x = 1.407 \text{ Mg m}^{-3}$

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

Cell parameters from 2447 reflections

$\theta = 3.5\text{--}28.1^\circ$

$\mu = 0.10 \text{ mm}^{-1}$

$T = 200 \text{ K}$

Block, brown

$0.29 \times 0.25 \times 0.13 \text{ mm}$

Z = 4

Data collection

| | |
|--|--|
| Bruker APEXII CCD diffractometer | 1008 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{\text{int}} = 0.031$ |
| graphite | $\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 1.8^\circ$ |
| φ and ω scans | $h = -15 \rightarrow 16$ |
| 4820 measured reflections | $k = -5 \rightarrow 3$ |
| 1289 independent reflections | $l = -15 \rightarrow 15$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.036$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.117$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.13$ | $w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.0455P]$ |
| 1289 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 81 parameters | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 0 restraints | $\Delta\rho_{\text{max}} = 0.29 \text{ e } \text{\AA}^{-3}$ |
| 0 constraints | $\Delta\rho_{\text{min}} = -0.22 \text{ e } \text{\AA}^{-3}$ |

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|------------|--------------|----------------------------------|
| O1 | 0.36008 (7) | 0.7180 (3) | 0.48635 (7) | 0.0334 (3) |
| H1 | 0.3249 | 0.7262 | 0.5328 | 0.050* |
| N1 | 0.26183 (8) | 0.7983 (3) | 0.15240 (8) | 0.0262 (3) |
| N2 | 0.43131 (8) | 0.5713 (3) | 0.30122 (10) | 0.0290 (3) |
| H71 | 0.4673 (14) | 0.495 (4) | 0.3773 (16) | 0.037 (4)* |
| H72 | 0.4404 (14) | 0.444 (5) | 0.2414 (17) | 0.051 (5)* |
| C1 | 0.32537 (9) | 0.7224 (3) | 0.27173 (10) | 0.0223 (3) |
| C2 | 0.28799 (9) | 0.8073 (3) | 0.36913 (10) | 0.0233 (3) |
| C3 | 0.18289 (10) | 0.9681 (3) | 0.33747 (11) | 0.0273 (3) |
| H3 | 0.1558 | 1.0290 | 0.4006 | 0.033* |
| C4 | 0.11580 (10) | 1.0419 (3) | 0.21157 (11) | 0.0302 (3) |
| H4 | 0.0424 | 1.1509 | 0.1877 | 0.036* |
| C5 | 0.15829 (10) | 0.9537 (3) | 0.12361 (10) | 0.0300 (3) |
| H5 | 0.1127 | 1.0043 | 0.0382 | 0.036* |

supplementary materials

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|------------|------------|------------|-------------|------------|-------------|
| O1 | 0.0280 (5) | 0.0579 (6) | 0.0161 (4) | 0.0062 (4) | 0.0109 (4) | 0.0035 (4) |
| N1 | 0.0277 (5) | 0.0355 (6) | 0.0170 (5) | -0.0017 (4) | 0.0103 (4) | 0.0006 (4) |
| N2 | 0.0275 (5) | 0.0420 (7) | 0.0207 (5) | 0.0060 (4) | 0.0129 (4) | 0.0020 (4) |
| C1 | 0.0237 (6) | 0.0271 (6) | 0.0182 (5) | -0.0031 (4) | 0.0106 (4) | -0.0002 (4) |
| C2 | 0.0246 (6) | 0.0300 (6) | 0.0167 (5) | -0.0025 (4) | 0.0096 (4) | -0.0004 (4) |
| C3 | 0.0284 (6) | 0.0337 (7) | 0.0235 (6) | -0.0001 (5) | 0.0143 (5) | -0.0026 (5) |
| C4 | 0.0251 (6) | 0.0360 (7) | 0.0297 (6) | 0.0045 (5) | 0.0109 (5) | 0.0028 (5) |
| C5 | 0.0280 (6) | 0.0399 (7) | 0.0197 (5) | 0.0003 (5) | 0.0069 (4) | 0.0047 (5) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-------------|--------------|-------------|--------------|
| O1—C2 | 1.3496 (13) | C1—C2 | 1.4219 (14) |
| O1—H1 | 0.8200 | C2—C3 | 1.3712 (16) |
| N1—C1 | 1.3312 (14) | C3—C4 | 1.3994 (16) |
| N1—C5 | 1.3490 (15) | C3—H3 | 0.9500 |
| N2—C1 | 1.3667 (14) | C4—C5 | 1.3675 (16) |
| N2—H71 | 0.870 (17) | C4—H4 | 0.9500 |
| N2—H72 | 0.895 (18) | C5—H5 | 0.9500 |
| C2—O1—H1 | 109.5 | C3—C2—C1 | 118.34 (10) |
| C1—N1—C5 | 118.74 (9) | C2—C3—C4 | 119.53 (10) |
| C1—N2—H71 | 117.7 (9) | C2—C3—H3 | 120.2 |
| C1—N2—H72 | 116.8 (11) | C4—C3—H3 | 120.2 |
| H71—N2—H72 | 115.2 (15) | C5—C4—C3 | 118.52 (11) |
| N1—C1—N2 | 118.74 (9) | C5—C4—H4 | 120.7 |
| N1—C1—C2 | 121.74 (10) | C3—C4—H4 | 120.7 |
| N2—C1—C2 | 119.46 (10) | N1—C5—C4 | 123.11 (10) |
| O1—C2—C3 | 125.40 (9) | N1—C5—H5 | 118.4 |
| O1—C2—C1 | 116.25 (10) | C4—C5—H5 | 118.4 |
| C5—N1—C1—N2 | -178.69 (10) | O1—C2—C3—C4 | -179.09 (11) |
| C5—N1—C1—C2 | -1.36 (18) | C1—C2—C3—C4 | 0.32 (18) |
| N1—C1—C2—O1 | -179.79 (10) | C2—C3—C4—C5 | -0.72 (19) |
| N2—C1—C2—O1 | -2.49 (17) | C1—N1—C5—C4 | 0.94 (19) |
| N1—C1—C2—C3 | 0.74 (18) | C3—C4—C5—N1 | 0.1 (2) |
| N2—C1—C2—C3 | 178.05 (11) | | |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|------------|-------------|-------------|---------------|
| O1—H1 \cdots N1 ⁱ | 0.82 | 1.85 | 2.6639 (12) | 172. |
| N2—H71 \cdots O1 ⁱⁱ | 0.870 (17) | 2.276 (17) | 3.0184 (13) | 143.2 (12) |
| N2—H72 \cdots N2 ⁱⁱⁱ | 0.895 (18) | 2.358 (17) | 3.1249 (15) | 143.8 (15) |

Symmetry codes: (i) $x, -y+3/2, z+1/2$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, y-1/2, -z+1/2$.

Fig. 1

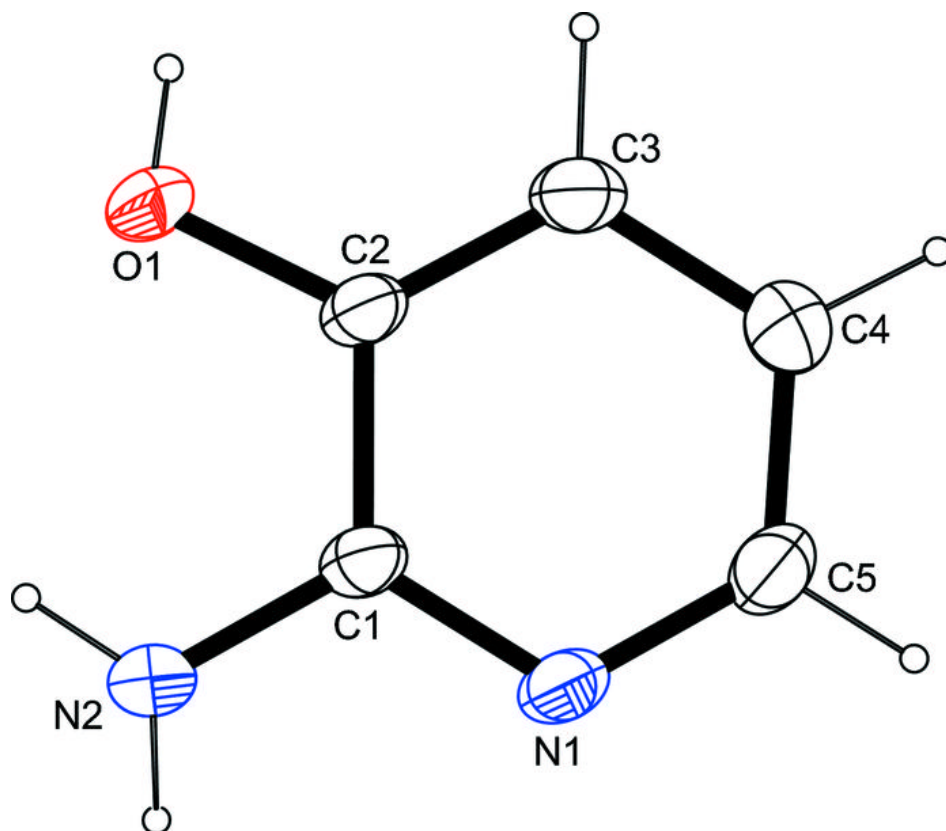


Fig. 2

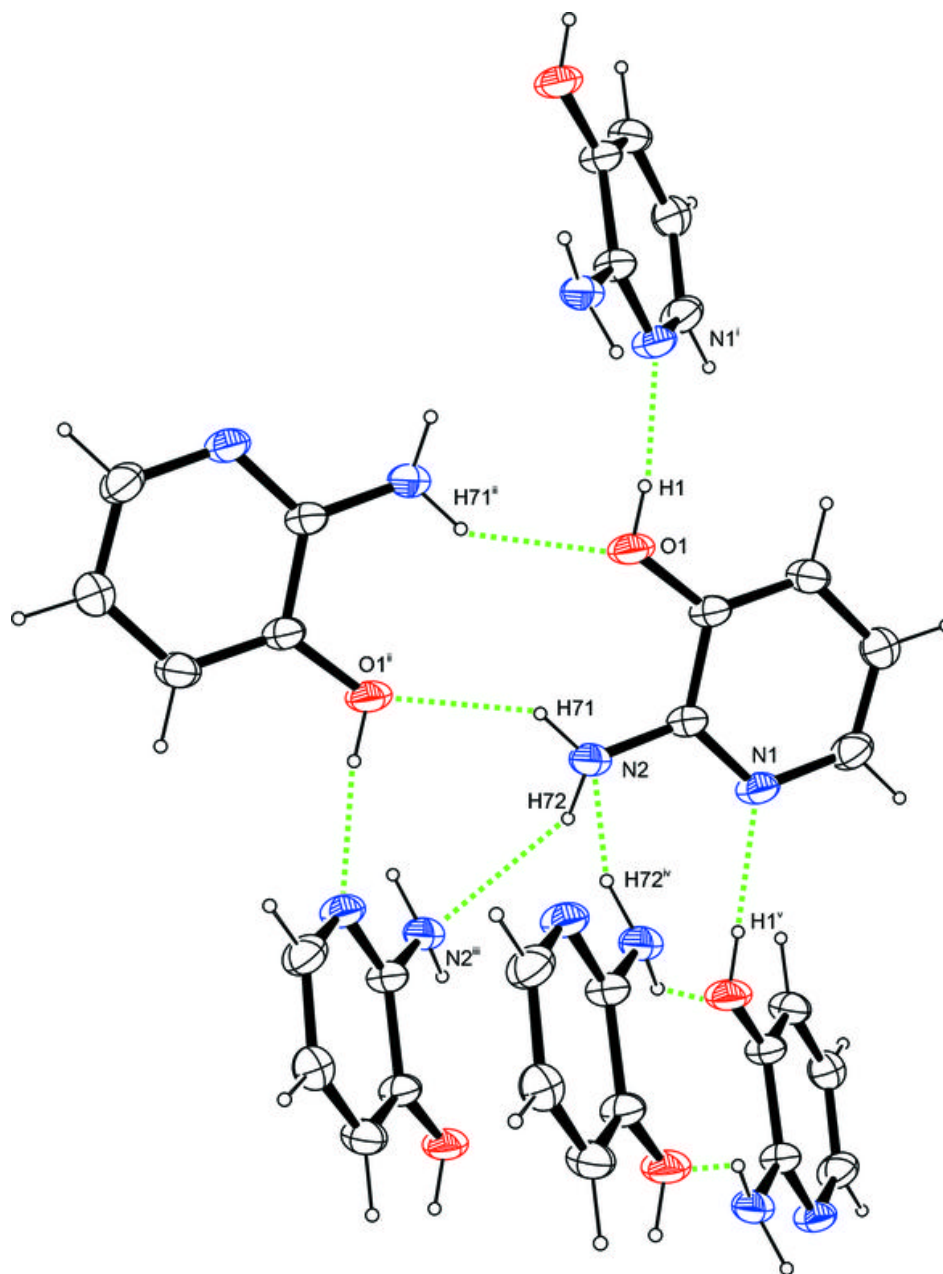


Fig. 3

