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2,4,5-Tri-4-pyridyl-1H-imidazole monohydrate

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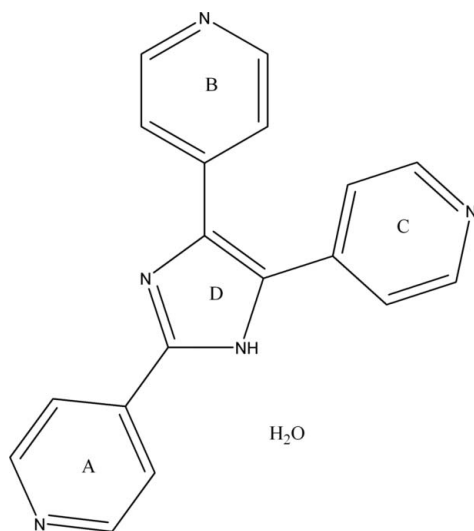
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
 R factor = 0.045; wR factor = 0.059; data-to-parameter ratio = 14.1.

The title compound, $\text{C}_{18}\text{H}_{13}\text{N}_5 \cdot \text{H}_2\text{O}$, was synthesized by the condensation of pyridine-4-carbaldehyde and ammonium acetate, forming a multipyridyl ligand. In the crystal, molecules are linked into chains by $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonds. The chains are linked by weak $\text{C}-\text{H} \cdots \text{N}$ interactions, generating a layer structure.

Related literature

2,4,5-Tri-4-pyridyl-imidazole is used in the construction of metal-organic coordination polymers, see: Liang *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{18}\text{H}_{13}\text{N}_5 \cdot \text{H}_2\text{O}$
 $M_r = 317.35$
 Triclinic, $P\bar{1}$
 $a = 8.910$ (2) Å
 $b = 9.401$ (2) Å
 $c = 10.638$ (2) Å

 $\alpha = 72.027$ (4)°
 $\beta = 70.624$ (4)°
 $\gamma = 77.716$ (4)°
 $V = 793.4$ (3) Å³
 $Z = 2$

 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.30 \times 0.26 \times 0.22$ mm

Data collection

 Bruker APEXII area-detector
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2004)
 $T_{\min} = 0.974$, $T_{\max} = 0.981$

 4313 measured reflections
 3067 independent reflections
 1720 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.059$
 $S = 1.02$
 3067 reflections
 217 parameters

 3 restraints
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.15$ e Å⁻³
 $\Delta\rho_{\min} = -0.15$ e Å⁻³

Table 1

Selected torsion angles (°).

C4—C3—C6—N2	−12.2 (4)	N1—C8—C9—C13	−88.5 (3)
C14—C7—C8—C9	1.8 (5)	N2—C7—C14—C18	−7.7 (3)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
N1—H1C \cdots O1	0.94	1.82	2.756 (2)	173
C10—H10 \cdots N2 ⁱ	0.93	2.59	3.467 (3)	158
O1—H1B \cdots N4 ⁱⁱ	0.91	1.96	2.869 (2)	174
O1—H1A \cdots N5 ⁱⁱⁱ	0.87	1.94	2.808 (2)	174

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

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXL97; software used to prepare material for publication: SHELXL97 and publCIF (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2553).

References

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 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Westrip, S. P. (2009). *publCIF*. In preparation.

supplementary materials

Acta Cryst. (2009). E65, o2329 [doi:10.1107/S1600536809032267]

2,4,5-Tri-4-pyridyl-1*H*-imidazole monohydrate

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Comment

2,4,5-tri(4-pyridyl)imidazole is a multipyridyl compound, which is useful to construct new metal-organic coordination polymers (Liang *et al.*, 2009). In this paper, we report the synthesis and X-ray crystal structure analysis of the title compound, (I), 2,4,5-tri(4-pyridyl)imidazole with one co-crystallized water molecule.

In 2,4,5-tri(4-pyridyl)imidazole three pyridyl groups are directly connected with the imidazole ring. The dihedral angles between the mean planes of pyridyl ring A and imidazole ring D is 11.6 (4)°, that of pyridyl ring B and imidazole ring D is 8.4 (3)°, and that of pyridyl ring C and imidazole ring D is 84.1 (3)°, suggesting that the plane of ring A and B are co-planar with ring D, but that ring C and ring D are almost vertical.

In the crystal lattice the molecules are linked by O—H···N hydrogen bonds, and by weak C—H···N interactions to generate a three-dimensional layer structure (Fig 2).

Experimental

A mixture of 2 g (0.018 mol) of 4-pyridinecarbaldehyde and 8 g (0.1 mol) of ammonium acetate was heated to 393 K with stirring 3 h. The reaction mixture was cooled, the precipitate was filtered off, washed with water, 5% solution of NaOH, and recrystallized from ethanol. Single crystals of 2,4,5-tri(4-pyridyl)imidazole suitable for X-ray analysis were obtained by slow evaporation at room temperature of a methanol solution. ¹H NMR (500 MHz, DMSO-*d*₆) 8.70(t, 4H), 8.54 (s, 2H), 8.02 (s, 2H), 7.53 (s, 4H) MS: found [*M*₊] = 299.1, cal [*M*₊] = 299.3.

Refinement

The H atoms of the pyridyl rings were constrained as idealized aromatic CH groups. The H atoms of water, H1A and H1B, were located in a difference Fourier map and the O1—H1A and O1—H1B were restrained to 0.85Å, the H1A—H1B was restrained to 1.35Å. The proton on the imidazole N atom, H1C, was also located in a difference Fourier map and N1—H1C was restrained to 0.94Å. The *U*_{iso}(H) was equal to 1.2 times that of the parent atoms for all H atoms.

Figures

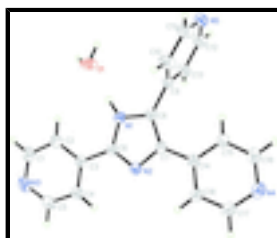


Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids.

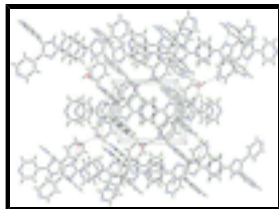


Fig. 2. The packing diagram of (I), viewed along the c axis; hydrogen bonds are shown as dashed lines.

2,4,5-Tri-4-pyridyl-1H-imidazole monohydrate

Crystal data

$C_{18}H_{13}N_5 \cdot H_2O$	$Z = 2$
$M_r = 317.35$	$F_{000} = 332$
Triclinic, $P\bar{1}$	$D_x = 1.328 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 8.910 (2) \text{ \AA}$	Cell parameters from 4826 reflections
$b = 9.401 (2) \text{ \AA}$	$\theta = 0.9\text{--}28.3^\circ$
$c = 10.638 (2) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\alpha = 72.027 (4)^\circ$	$T = 293 \text{ K}$
$\beta = 70.624 (4)^\circ$	Block, yellow
$\gamma = 77.716 (4)^\circ$	$0.30 \times 0.26 \times 0.22 \text{ mm}$
$V = 793.4 (3) \text{ \AA}^3$	

Data collection

Bruker APEXII area-detector diffractometer	3067 independent reflections
Radiation source: fine-focus sealed tube	1720 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.025$
$T = 293 \text{ K}$	$\theta_{\text{max}} = 26.1^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$h = -11 \rightarrow 10$
$T_{\text{min}} = 0.974$, $T_{\text{max}} = 0.981$	$k = -11 \rightarrow 8$
4313 measured reflections	$l = -13 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H-atom parameters constrained
$wR(F^2) = 0.059$	$w = 1/[\sigma^2(F_o^2) + (0.0004P)^2]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
3067 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
217 parameters	$\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$

3 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3675 (3)	0.6040 (2)	0.2766 (3)	0.0664 (8)
H1	0.2702	0.6621	0.2711	0.080*
C2	0.3638 (3)	0.4768 (2)	0.3848 (2)	0.0539 (7)
H2	0.2669	0.4515	0.4492	0.065*
C3	0.5060 (3)	0.3871 (2)	0.3965 (2)	0.0403 (6)
C4	0.6444 (3)	0.4344 (2)	0.2983 (2)	0.0505 (7)
H4	0.7436	0.3798	0.3023	0.061*
C5	0.6343 (3)	0.5632 (3)	0.1943 (2)	0.0616 (8)
H5	0.7295	0.5920	0.1291	0.074*
C6	0.5121 (2)	0.2487 (2)	0.5058 (2)	0.0391 (6)
C7	0.5929 (2)	0.0342 (2)	0.6266 (2)	0.0394 (6)
C8	0.4352 (2)	0.0717 (2)	0.6957 (2)	0.0409 (6)
C9	0.3254 (2)	-0.0054 (2)	0.8263 (2)	0.0411 (6)
C10	0.2358 (3)	-0.1086 (2)	0.8270 (3)	0.0599 (8)
H10	0.2408	-0.1288	0.7453	0.072*
C11	0.1383 (3)	-0.1815 (3)	0.9518 (3)	0.0648 (8)
H11	0.0798	-0.2522	0.9513	0.078*
C12	0.2082 (3)	-0.0560 (3)	1.0683 (3)	0.0626 (8)
H12	0.1995	-0.0363	1.1511	0.075*
C13	0.3095 (2)	0.0224 (2)	0.9487 (2)	0.0530 (7)
H13	0.3659	0.0932	0.9519	0.064*
C14	0.7046 (2)	-0.1000 (2)	0.6596 (2)	0.0408 (6)
C15	0.6737 (2)	-0.2114 (2)	0.7826 (2)	0.0518 (7)
H15	0.5777	-0.2024	0.8512	0.062*
C16	0.7863 (3)	-0.3359 (2)	0.8028 (3)	0.0609 (8)
H16	0.7620	-0.4090	0.8862	0.073*
C17	0.9555 (3)	-0.2501 (3)	0.5936 (3)	0.0675 (9)
H17	1.0532	-0.2614	0.5274	0.081*
C18	0.8497 (2)	-0.1228 (2)	0.5640 (2)	0.0558 (8)
H18	0.8765	-0.0523	0.4792	0.067*

supplementary materials

N1	0.38651 (19)	0.20619 (17)	0.61671 (17)	0.0441 (5)
H1C	0.2849	0.2595	0.6454	0.053*
N2	0.63900 (18)	0.14695 (18)	0.50748 (17)	0.0417 (5)
N3	0.4987 (3)	0.6500 (2)	0.1798 (2)	0.0669 (7)
N4	0.9263 (2)	-0.3581 (2)	0.7116 (2)	0.0642 (7)
N5	0.1230 (2)	-0.1573 (2)	1.0716 (2)	0.0578 (6)
O1	0.07758 (16)	0.34217 (15)	0.70240 (16)	0.0723 (6)
H1A	0.0112	0.2912	0.7740	0.087*
H1B	0.0296	0.4357	0.7119	0.087*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0606 (18)	0.0525 (17)	0.075 (2)	-0.0030 (13)	-0.0288 (16)	0.0057 (15)
C2	0.0493 (16)	0.0470 (16)	0.0557 (18)	-0.0069 (12)	-0.0138 (13)	-0.0002 (13)
C3	0.0441 (14)	0.0339 (13)	0.0413 (15)	-0.0052 (11)	-0.0110 (12)	-0.0087 (11)
C4	0.0506 (15)	0.0445 (15)	0.0472 (16)	-0.0069 (12)	-0.0090 (13)	-0.0039 (13)
C5	0.0673 (19)	0.0564 (17)	0.0499 (18)	-0.0210 (14)	-0.0076 (15)	0.0009 (14)
C6	0.0371 (14)	0.0352 (13)	0.0391 (15)	-0.0044 (11)	-0.0068 (12)	-0.0059 (11)
C7	0.0389 (14)	0.0316 (13)	0.0431 (15)	-0.0020 (10)	-0.0094 (11)	-0.0072 (12)
C8	0.0427 (14)	0.0306 (13)	0.0403 (15)	-0.0023 (11)	-0.0074 (12)	-0.0029 (11)
C9	0.0379 (14)	0.0310 (14)	0.0423 (16)	0.0039 (10)	-0.0062 (12)	-0.0035 (12)
C10	0.0671 (18)	0.0481 (16)	0.0545 (18)	-0.0157 (13)	0.0014 (14)	-0.0138 (14)
C11	0.0677 (19)	0.0475 (17)	0.069 (2)	-0.0186 (13)	-0.0037 (17)	-0.0105 (16)
C12	0.0691 (19)	0.0614 (18)	0.0475 (18)	-0.0080 (14)	-0.0121 (15)	-0.0054 (15)
C13	0.0559 (17)	0.0472 (16)	0.0466 (17)	-0.0126 (12)	-0.0087 (13)	-0.0017 (13)
C14	0.0371 (13)	0.0352 (13)	0.0478 (16)	-0.0029 (10)	-0.0111 (12)	-0.0097 (12)
C15	0.0468 (15)	0.0437 (15)	0.0510 (17)	0.0046 (12)	-0.0081 (13)	-0.0060 (13)
C16	0.0644 (18)	0.0460 (16)	0.0592 (19)	0.0018 (14)	-0.0175 (15)	-0.0014 (13)
C17	0.0456 (16)	0.0552 (18)	0.077 (2)	0.0033 (13)	-0.0018 (15)	-0.0065 (16)
C18	0.0424 (14)	0.0429 (15)	0.0631 (18)	0.0009 (12)	-0.0061 (13)	-0.0009 (13)
N1	0.0363 (11)	0.0334 (11)	0.0475 (13)	0.0033 (8)	-0.0024 (10)	-0.0050 (10)
N2	0.0382 (11)	0.0356 (11)	0.0430 (12)	-0.0028 (9)	-0.0068 (9)	-0.0048 (9)
N3	0.0751 (16)	0.0556 (14)	0.0610 (16)	-0.0149 (13)	-0.0254 (13)	0.0083 (12)
N4	0.0509 (14)	0.0481 (13)	0.0766 (17)	0.0070 (10)	-0.0139 (12)	-0.0061 (12)
N5	0.0524 (14)	0.0451 (14)	0.0571 (16)	-0.0041 (10)	-0.0046 (12)	-0.0001 (12)
O1	0.0484 (10)	0.0450 (10)	0.0835 (13)	0.0044 (7)	0.0126 (9)	-0.0020 (9)

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

C1—N3	1.325 (2)	C10—H10	0.9300
C1—C2	1.377 (3)	C11—N5	1.321 (3)
C1—H1	0.9300	C11—H11	0.9300
C2—C3	1.385 (3)	C12—N5	1.324 (3)
C2—H2	0.9300	C12—C13	1.385 (3)
C3—C4	1.379 (2)	C12—H12	0.9300
C3—C6	1.458 (3)	C13—H13	0.9300
C4—C5	1.374 (3)	C14—C18	1.377 (2)
C4—H4	0.9300	C14—C15	1.386 (3)

C5—N3	1.333 (3)	C15—C16	1.382 (3)
C5—H5	0.9300	C15—H15	0.9300
C6—N2	1.318 (2)	C16—N4	1.324 (2)
C6—N1	1.352 (2)	C16—H16	0.9300
C7—C8	1.379 (2)	C17—N4	1.335 (3)
C7—N2	1.381 (2)	C17—C18	1.380 (3)
C7—C14	1.460 (3)	C17—H17	0.9300
C8—N1	1.359 (2)	C18—H18	0.9300
C8—C9	1.480 (3)	N1—H1C	0.9393
C9—C13	1.362 (3)	O1—H1A	0.8697
C9—C10	1.378 (3)	O1—H1B	0.9124
C10—C11	1.383 (3)		
N3—C1—C2	125.1 (2)	N5—C11—H11	117.9
N3—C1—H1	117.4	C10—C11—H11	117.9
C2—C1—H1	117.4	N5—C12—C13	123.8 (3)
C1—C2—C3	119.2 (2)	N5—C12—H12	118.1
C1—C2—H2	120.4	C13—C12—H12	118.1
C3—C2—H2	120.4	C9—C13—C12	119.2 (2)
C4—C3—C2	116.7 (2)	C9—C13—H13	120.4
C4—C3—C6	120.74 (19)	C12—C13—H13	120.4
C2—C3—C6	122.61 (19)	C18—C14—C15	116.15 (19)
C5—C4—C3	119.3 (2)	C18—C14—C7	119.69 (19)
C5—C4—H4	120.3	C15—C14—C7	124.15 (19)
C3—C4—H4	120.3	C16—C15—C14	119.7 (2)
N3—C5—C4	125.0 (2)	C16—C15—H15	120.1
N3—C5—H5	117.5	C14—C15—H15	120.1
C4—C5—H5	117.5	N4—C16—C15	124.4 (2)
N2—C6—N1	111.24 (18)	N4—C16—H16	117.8
N2—C6—C3	124.49 (18)	C15—C16—H16	117.8
N1—C6—C3	124.24 (18)	N4—C17—C18	124.0 (2)
C8—C7—N2	109.24 (17)	N4—C17—H17	118.0
C8—C7—C14	130.29 (19)	C18—C17—H17	118.0
N2—C7—C14	120.44 (17)	C14—C18—C17	120.1 (2)
N1—C8—C7	105.72 (17)	C14—C18—H18	119.9
N1—C8—C9	121.56 (17)	C17—C18—H18	119.9
C7—C8—C9	132.71 (19)	C6—N1—C8	108.05 (16)
C13—C9—C10	118.0 (2)	C6—N1—H1C	129.5
C13—C9—C8	121.6 (2)	C8—N1—H1C	122.1
C10—C9—C8	120.4 (2)	C6—N2—C7	105.72 (16)
C9—C10—C11	118.5 (2)	C1—N3—C5	114.6 (2)
C9—C10—H10	120.7	C16—N4—C17	115.58 (19)
C11—C10—H10	120.7	C11—N5—C12	116.2 (2)
N5—C11—C10	124.3 (3)	H1A—O1—H1B	96.8
N3—C1—C2—C3	-0.1 (4)	C8—C7—C14—C18	170.3 (3)
C1—C2—C3—C4	1.3 (4)	N2—C7—C14—C18	-7.7 (3)
C1—C2—C3—C6	-178.4 (2)	C8—C7—C14—C15	-8.4 (4)
C2—C3—C4—C5	-1.5 (3)	N2—C7—C14—C15	173.6 (2)
C6—C3—C4—C5	178.2 (2)	C18—C14—C15—C16	0.0 (4)

supplementary materials

C3—C4—C5—N3	0.5 (4)	C7—C14—C15—C16	178.7 (2)
C4—C3—C6—N2	-12.2 (4)	C14—C15—C16—N4	0.3 (4)
C2—C3—C6—N2	167.5 (2)	C15—C14—C18—C17	-0.6 (4)
C4—C3—C6—N1	169.8 (2)	C7—C14—C18—C17	-179.4 (2)
C2—C3—C6—N1	-10.4 (4)	N4—C17—C18—C14	0.9 (4)
N2—C7—C8—N1	0.8 (3)	N2—C6—N1—C8	1.3 (3)
C14—C7—C8—N1	-177.4 (2)	C3—C6—N1—C8	179.5 (2)
N2—C7—C8—C9	180.0 (2)	C7—C8—N1—C6	-1.3 (3)
C14—C7—C8—C9	1.8 (5)	C9—C8—N1—C6	179.5 (2)
N1—C8—C9—C13	-88.5 (3)	N1—C6—N2—C7	-0.8 (3)
C7—C8—C9—C13	92.5 (3)	C3—C6—N2—C7	-179.0 (2)
N1—C8—C9—C10	91.7 (3)	C8—C7—N2—C6	0.0 (3)
C7—C8—C9—C10	-87.4 (4)	C14—C7—N2—C6	178.4 (2)
C13—C9—C10—C11	-1.8 (3)	C2—C1—N3—C5	-0.9 (4)
C8—C9—C10—C11	178.05 (19)	C4—C5—N3—C1	0.7 (4)
C9—C10—C11—N5	1.1 (4)	C15—C16—N4—C17	0.0 (4)
C10—C9—C13—C12	1.6 (3)	C18—C17—N4—C16	-0.6 (4)
C8—C9—C13—C12	-178.25 (19)	C10—C11—N5—C12	0.0 (4)
N5—C12—C13—C9	-0.6 (4)	C13—C12—N5—C11	-0.2 (4)

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>
N1—H1C \cdots O1	0.94	1.82	2.756 (2)	173
C10—H10 \cdots N2 ⁱ	0.93	2.59	3.467 (3)	158
O1—H1B \cdots N4 ⁱⁱ	0.91	1.96	2.869 (2)	174
O1—H1A \cdots N5 ⁱⁱⁱ	0.87	1.94	2.808 (2)	174

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

Fig. 1

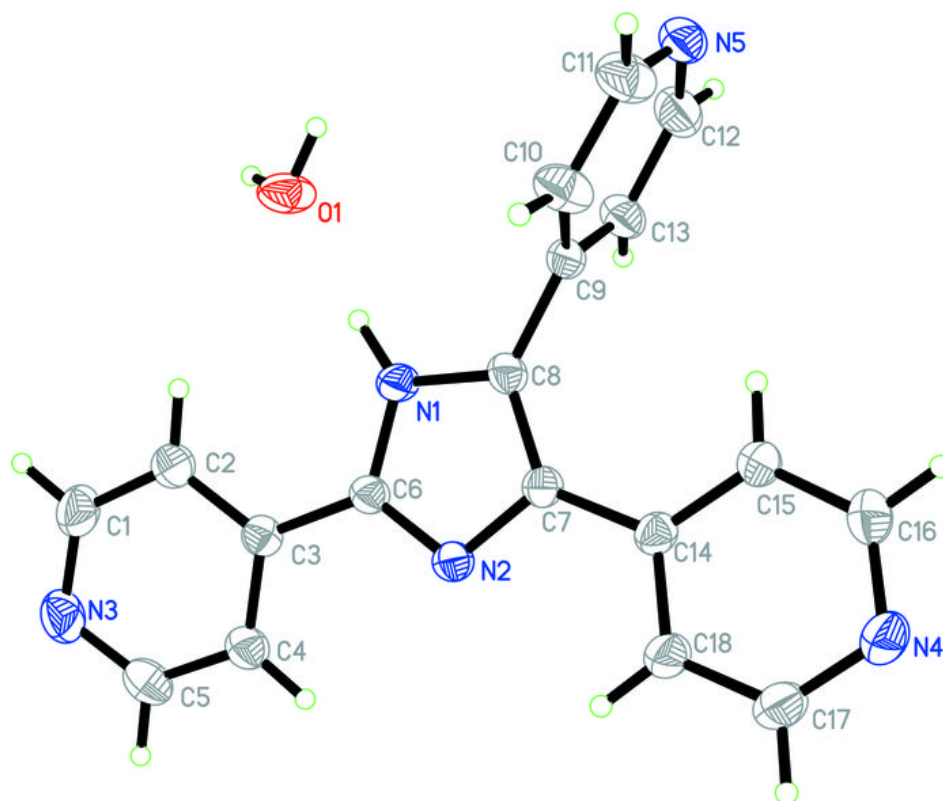


Fig. 2

