Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Di-2-pyridyl ketone *p*-aminobenzoylhydrazone hydrate

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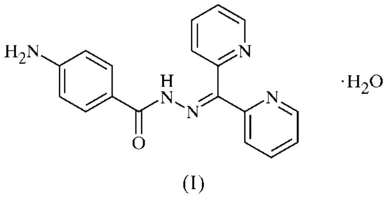
Received 12 February 2002
Accepted 8 March 2002
Online 11 April 2002

The title compound [systematic name: 4-amino-2′-(di-2-pyridylmethylene)benzohydrazide hydrate], $C_{18}H_{15}N_5O\cdot H_2O$, crystallizes in the triclinic space group $P\overline{1}$. Structural analysis shows one pyridine ring and the *p*-aminobenzoylhydrazone moiety to be coplanar and orthogonal to the second pyridine ring. The packing reveals infinite molecular units interlocked *via* a network of hydrogen bonds.

Comment

Hydrazones and their metal complexes have been studied with regard to their physical properties, reactivity patterns and application in a variety of processes, including non-linear optics and molecular sensing (Bakir *et al.*, 2000, Bakir, 2002*a*,*b*,*c*; Pan *et al.*, 1997). We have been interested in the chemistry of di-2-pyridyl ketone and its oxime and hydrazone derivatives, and have reported on the structures of Re compounds of the type *fac*-$Re(CO)_3(L\text{–}L)Cl$, where *L–L* is di-2-pyridyl ketone *p*-nitrophenylhydrazone (dpknph), di-2-pyridyl ketone 2,4-dinitrophenylhydrazone (dpkdnph), di-2-pyridyl ketone phenylhydrazone (dpkphh) or di-2-pyridyl oxime (dpkoxime) (Bakir & McKenzie, 1997*a*,*b*; Bakir & Abdur-Rashid, 1999; Bakir, 1999, 2001*a*,*b*, 2002*a*,*b*,*c*). Structural studies on the optical sensor di-2-pyridyl ketone benzoylhydrazone (dpkbz) revealed a network of non-covalent interactions that may account for its optosensing behaviour (Bakir & Brown, 2002). We report here on the structure of di-2-pyridyl ketone *p*-aminobenzoylhydrazone hydrate, dpkabz·H_2O, (I).



(I)

The molecular structure of (I) is shown in Fig. 1, and selected bond distances and angles are given in Table 1. The *p*-aminobenzoylhydrazone moiety and the N1 pyridine ring are coplanar and orthogonal to the N2 pyridine ring. The orthogonality of the pyridine rings is similar to what is observed in dpkhydrazones such as dpkbz and di-2-pyridyl ketone 2-pyridylhydrazone (dpkph) (Bakir & Brown, 2002; Ishak *et al.*, 1984). The bond distances and angles are normal, and similar to those reported for dpkbz and other related compounds. For example, in dpkbz, C═N, N—N and C═O bond distances of 1.30 (2), 1.36 (2) and 1.22 (2) Å, respectively, and N—N═C, O═C—N and O═C—C bond angles of 120.6 (2), 123.6 (2) and 122.2 (2)°, respectively, were reported by Bakir & Brown (2002).

The packing of the molecules of (I) (Fig. 2) shows a web of dpkabz·H_2O units interlocked *via* a network of classical and non-classical hydrogen bonds (Fig. 3 and Table 2). The coordination about the O atom of the solvated water molecule is tetrahedral (Fig. 3*a*), with two H atoms from the *p*-amino groups and the two water H atoms occupying the coordination around the O atom. Each solvated water molecule interlocks four dpkabz molecules through a web of classical hydrogen bonds of the type O—H···*X*, where *X* is O or N (Table 2 and

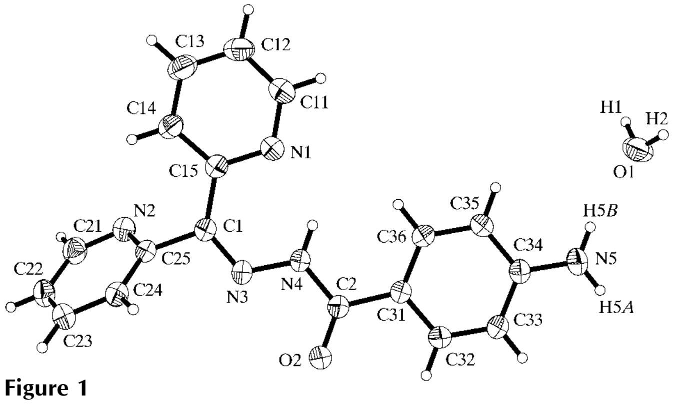


Figure 1
A view of the molecule of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

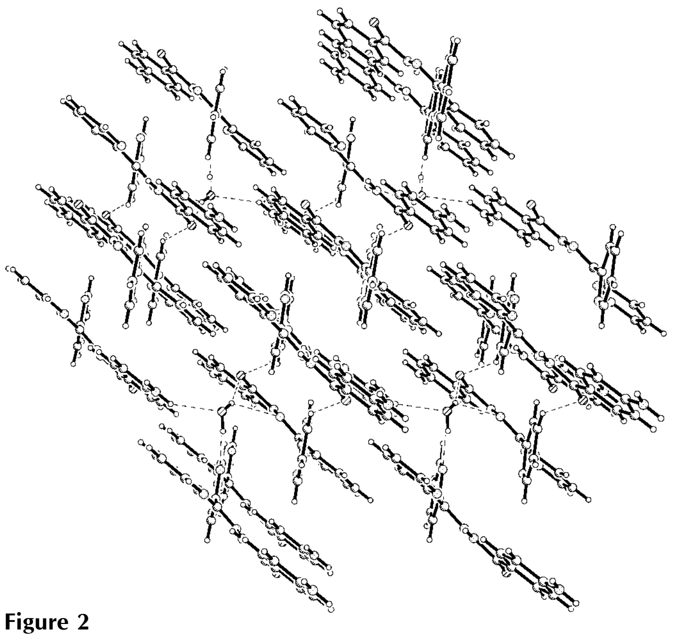


Figure 2
A packing diagram for (I). Hydrogen bonds are shown by dashed lines.

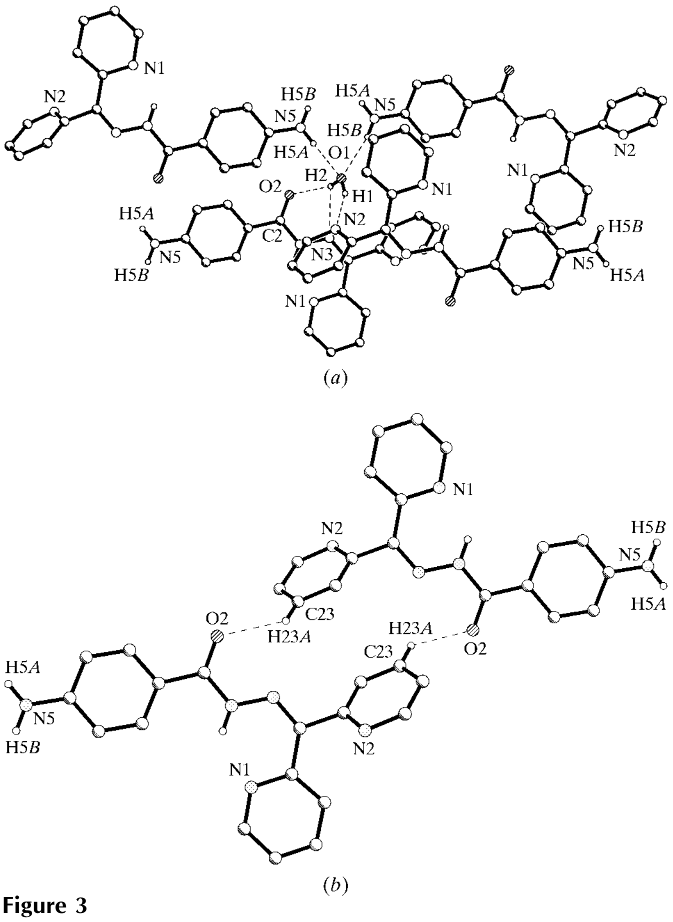
DOI: 10.1107/S0108270102004511

Fig. 3*a*), and each dpkabz molecule is hydrogen bonded to adjacent dpkabz molecules through a network of non-classical hydrogen bonds of the type O···H—C (Fig. 3*b*). The bond distances and angles of these hydrogen bonds are of the same order as those reported for dpkbzh and *fac*-Re(CO)$_3$-(dpknph)Cl·dmso (dmso is dimethyl sulfoxide) and other compounds containing such bonds (Bakir, 2001*b*; Bakir & Brown, 2002; Braga *et al.*, 1998; Glusker *et al.*, 1994). For example, hydrogen-bond parameters of 0.82, 1.88 and 2.68 Å, and 164° were reported for O···H—O in *fac*-Re(CO)$_3$(dpkO,OH)Cl [dpkO,OH is hydroxybis(2-pyridyl)methanolate; Bakir, 2002*c*], and parameters of 1.00, 2.50 and 3.24 Å, and 130.6° were observed for the soft non-classical C—H···O hydrogen bond in dpkbz (Bakir & Brown, 2002).

Due to their convenient synthesis, rich physicochemical properties and application in a variety of processes, work is in progress to prepare a series of di-2-pyridyl ketone hydrazones and their metal compounds to explore their solid-state structures and electro-optical properties.

Experimental

Dpkabz was synthesized by the reaction of *p*-aminobenzhydrazide with di-2-pyridyl ketone in refluxing ethanol/HCl solution, using a procedure similar to that reported for the synthesis of dpknph by Bakir & Abdur-Rashid (1999). When dpkabz was allowed to stand in an ethanol/hexanes solution for several days at room temperature, yellow–brown crystals of dpkabz·H$_2$O, (I), were obtained. A single crystal was selected and mounted on a glass fibre using epoxy cement, and used for data collection.



(*a*)

(*b*)

Figure 3
Views of the hydrogen bonds in (I). Table 2 gives details of the symmetry codes used to generate equivalent atoms. All aromatic H atoms have been omitted for clarity.

Crystal data

$C_{18}H_{15}N_5O{\cdot}H_2O$
$M_r = 335.37$
Triclinic, $P\overline{1}$
$a = 8.6648$ (10) Å
$b = 9.9630$ (16) Å
$c = 11.1996$ (11) Å
$\alpha = 91.083$ (9)°
$\beta = 109.199$ (5)°
$\gamma = 109.571$ (10)°
$V = 851.21$ (19) Å^3

$Z = 2$
$D_x = 1.308$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 46 reflections
$\theta = 3.9$–12.4°
$\mu = 0.09$ mm^{-1}
$T = 298$ (2) K
Octahedral, yellow–brown
0.40 × 0.25 × 0.10 mm

Data collection

Bruker *P*4 diffractometer
$2\theta/\omega$ scans
Absorption correction: empirical *via* ψ scans (*XSCANS*; Bruker, 1996)
$T_{\min} = 0.957$, $T_{\max} = 0.991$
7103 measured reflections
6067 independent reflections
1820 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.029$
$\theta_{\max} = 32.5°$
$h = -1 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 16$
3 standard reflections every 97 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.124$
$S = 0.95$
6067 reflections
235 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0156 (15)

Table 1
Selected geometric parameters (Å, °).

C15—C1	1.478 (2)	N4—C2	1.3675 (19)
C1—N3	1.2957 (19)	C2—O2	1.2260 (18)
C1—C25	1.492 (2)	C2—C31	1.472 (2)
N3—N4	1.3643 (16)	C34—N5	1.3668 (19)
C15—N1—C11	117.43 (16)	N2—C21—C22	123.7 (2)
N1—C11—C12	124.1 (2)	C1—N3—N4	119.37 (14)
N3—C1—C15	128.82 (14)	N3—N4—C2	117.83 (13)
N3—C1—C25	111.42 (14)	O2—C2—N4	120.86 (15)
C15—C1—C25	119.76 (14)	O2—C2—C31	122.73 (15)
C25—N2—C21	116.57 (17)	N4—C2—C31	116.37 (15)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···N1	0.86	2.01	2.656 (2)	131
N5—H5*B*···O1	0.86	2.13	2.979 (2)	168
C14—H14*A*···N2	0.93	2.57	3.048 (2)	112
N5—H5*A*···O1$^{\rm i}$	0.86	2.32	3.086 (2)	149
O1—H1···N2$^{\rm ii}$	0.90 (3)	2.03 (3)	2.880 (3)	158 (2)
O1—H2···O2$^{\rm iii}$	0.83 (3)	2.26 (3)	3.043 (2)	158 (3)
O1—H2···N3$^{\rm iii}$	0.83 (3)	2.57 (3)	3.193 (2)	133 (3)
C23—H23*A*···O2$^{\rm iv}$	0.93	2.55	3.325 (2)	142

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

With the exception of atoms H1 and H2, which were assigned from a difference Fourier map and refined freely, all H atoms were assigned by assuming idealized geometry, with C—H = 0.93 Å and N—H = 0.86 Å.

Data collection: *XSCANS* (Bruker, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors acknowledge The University of the West Indies, the Inter-America Development Bank for funds to establish the X-ray laboratory at The University of the West Indies, Mona Campus, Jamaica, and Dr I. Hassan for technical assistance.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1194). Services for accessing these data are described at the back of the journal.

References

Bakir, M. (1999). *J. Electroanal. Chem.* **466**, 60–66.
Bakir, M. (2001*a*). *Acta Cryst.* C**57**, 1154–1156.
Bakir, M. (2001*b*). *Acta Cryst.* C**57**, 1371–1373.
Bakir, M. (2002*a*). *Eur. J. Inorg. Chem.* pp. 481–486.
Bakir, M. (2002*b*). *Inorg. Chim. Acta*, **332**, 1–6.
Bakir, M. (2002*c*). *Acta Cryst.* C**58**, m74–m76.
Bakir, M. & Abdur-Rashid, K. (1999). *Transition Met. Chem.* **24**, 384–388.
Bakir, M., Abdur-Rashid, K. & Mulder, W. H. (2000). *Talanta*, **51**, 735–741.
Bakir, M. & Brown, O. (2002). *J. Mol. Struct.* **609**, 129–136.
Bakir, M. & McKenzie, J. A. M. (1997*a*). *J. Electroanal. Chem.* **425**, 61–66.
Bakir, M. & McKenzie, J. A. M. (1997*b*). *J. Chem. Soc. Dalton Trans.* pp. 3571–3578.
Braga, D., Grepioni, F. & Desiraju, G. R. (1998). *Chem. Rev.* **98**, 1375–1405.
Bruker (1996). *XSCANS*. Version 2.2. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Glusker, J. P., Lewis, J. P. M. & Rossi, M. (1994). *Crystal Structure Analysis for Chemists and Biologists*, pp. 631–634. New York: Wiley–VCH.
Ishak, C. F., Pflaum, R. T. & Baenziger, N. C. (1984). *Acta Cryst.* C**40**, 2047–2049.
Pan, F., Bosshard, C., Wong, M. S., Serbutoviez, C., Schenk, K., Gramlich, V. & Gunter, P. (1997). *Chem. Mater.* **9**, 1328–1334.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.