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N-[6-(Hydroxymethyl)pyridin-2-yl]-2,2-dimethylpropanamide

Shyamaprosad Goswami,[a] Swapan Dey,[a] Suchada Chantrapromma[b] and Hoong-Kun Fun[c]*

[a]Department of Chemistry, Bengal Engineering and Science University, Shibpur, West Bengal 711103, India, [b]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail: hkfun@usm.my

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Key indicators

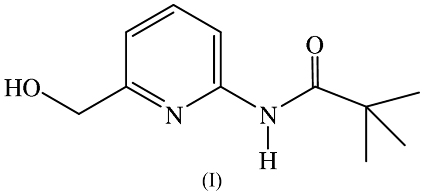
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.146
Data-to-parameter ratio = 15.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{11}H_{16}N_2O_2$, inversion-related molecules form an O—H···O and N—H···O hydrogen-bonded dimer. The hydrogen bonds generate rings of motifs $R_2^1(6)$ and $R_2^2(10)$. Weak π–π interactions between the dimers stabilize the crystal packing.

Comment

Hydrogen bonding and π stacking interactions are the two key forces in natural and artificial molecular recognition (Lehn, 1995; Jeffery & Saenger, 1995; Papadopoulou *et al.*, 1995; Hamilton, 1995). This concept is vital in the design of new-generation drugs, in new crystal material designs and in the creation of new artificial catalysts for chemical reactions. It is also an effective method of creating novel structures of molecular self-assemblies (Lehn *et al.*, 1992). We have previously reported (Goswami, Ghosh *et al.*, 1999; Goswami, Mahapatra *et al.*, 1999; Goswami, Ghosh & Dasgupta, 2000; Goswami *et al.*, 2001) the design and synthesis of a number of artificial receptors for molecular recognition of important biological substrates in the solution phase. We are also interested in the design of supramolecular structures in the solid phase, in search of novel motifs of hydrogen bonds in simple crystal engineering involving functionalized organic and heterocyclic compounds (Alkorta *et al.*, 2002; Alvarez-Rua *et al.*, 2004), including the new dimers of heterocycles. We report here the crystal structure of the title compound, (I).



(I)

The bond lengths and angles in (I) (Table 1) are within normal ranges (Allen *et al.*, 1987) and agree with the corresponding values in related structures (Goswami, Ghosh *et al.*, 1999; Goswami, Mahapatra *et al.*, 1999). The pyridine ring is planar within ±0.008 (2) Å (Fig. 1). The hydroxymethyl group is twisted away from the pyridine ring, the O1—C6—C5—C4 torsion angle being 112.2 (2)°. The mean plane through atoms N2, O2, C8 and C9 makes a dihedral angle of 6.5 (1)° with the pyridine plane.

The molecules are linked together to form a dimer by O1—H1*A*···N1^{i} and N2—H2*A*···O1^{i} hydrogen bonds [symmetry code: (i) $1 - x, 1 - y, 1 - z$], involving the hydroxy group and pyridine and amide N atoms (Table 2 and Fig. 2). In the dimer, the hydrogen bonds generate two $R_2^1(6)$ rings and one $R_2^2(10)$

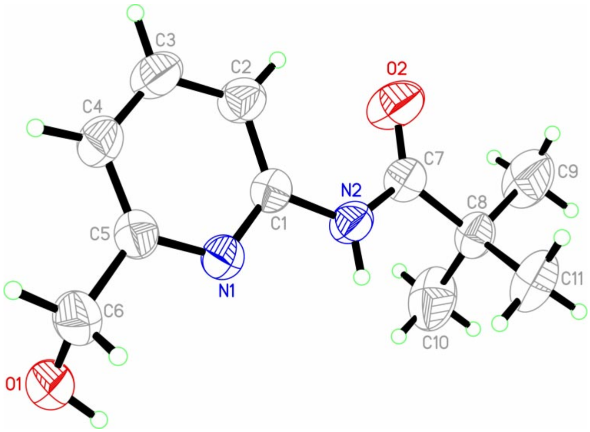


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

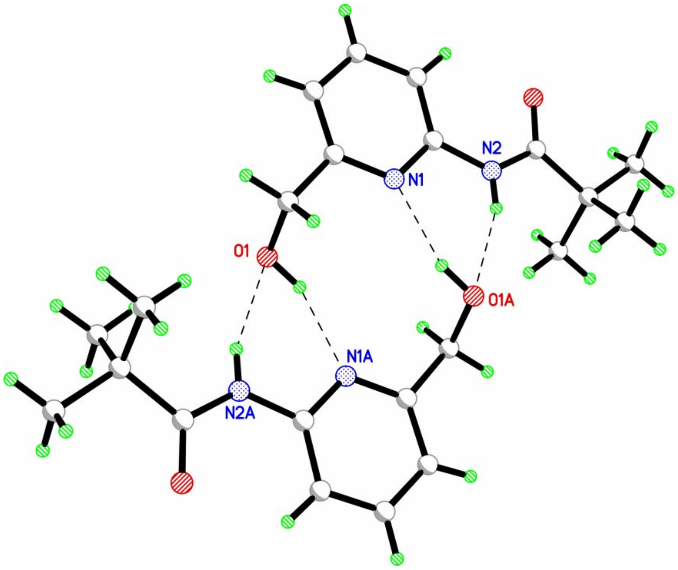


Figure 2
A view of the O—H···N and N—H···O hydrogen-bonded dimer. Atoms with the suffix *A* are generated by the symmetry operation (1 − *x*, 1 − *y*, 1 − *z*).

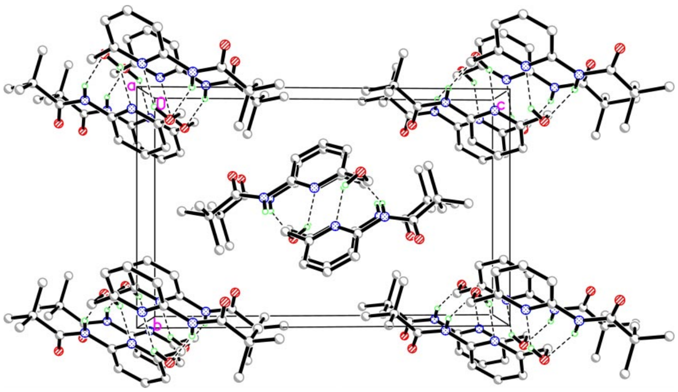


Figure 3
The packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonds have been omitted.

ring motif (Bernstein *et al.*, 1995). Interestingly, the hydroxy group is not involved in forming a five-membered intramolecular hydrogen-bonded ring by participation of the hydroxy group and pyridine N atom, but the hydroxy group in (I) acts as a hydrogen-bond donor (to the pyridine N atom) as well as an acceptor, with its lone pair (for the pyridine amide NH group) forming the dimer (Fig. 2). Thus each dimer is stabilized by forming two identical intermolecular hydrogen-bonded six-membered rings separated by a large ten-membered ring flexible enough to accommodate the benzylic methylene group. Interestingly, the formation of this type of hydrogen-bonded dimeric structure is also observed in the crystal structure of 2-acetylamino-6-methylpyridine *N*-oxide (Goswami, Ghosh *et al.*, 1999; Goswami, Ghosh & Mukherjee, 2000), where the two six-membered rings are formed by intermolecular hydrogen bonds involving the *N*-oxide, amide NH and methyl groups.

The stability in the molecular packing is achieved by the stacking interaction of the two pyridine rings. The dimers are arranged in such a way that the pyridine rings of the molecules at (*x*, *y*, *z*) and (−*x*, 1 − *y*, 1 − *z*) are stacked 3.488 (1) Å apart [centroid–centroid distance is 3.863 (1) Å], indicating weak π–π interactions (Fig. 3).

Experimental

2-Pivaloylamino-6-methylpyridine, a low melting solid, was mixed thoroughly with a stoichiometric amount of selenium dioxide (Goswami & Adak, 2003) in a conical flask and was oxidized under microwave radiation at 450 W for 25 min. The solid was dissolved in chloroform, filtered and passed through a pad of celite. After evaporation of the solvent in a rotary evaporator, the crude mixture (containing 2-pivaloylamino-6-formylpyridine as the major product along with 2-pivaloylamino-6-hydroxymethylpyridine as a minor product) was treated with $NaBH_4$ in ethanol to obtain the title compound, which was purified by silica gel column chromatography (m.p. 390–393 K). Single crystals were obtained by slow evaporation of a chloroform–methanol (95:5) solution.

Crystal data

$C_{11}H_{16}N_2O_2$
M_r = 208.26
Monoclinic, $P2_1/n$
a = 6.8330 (6) Å
b = 10.3313 (10) Å
c = 16.1635 (15) Å
β = 94.486 (2)°
V = 1137.55 (18) Å^3
Z = 4
D_x = 1.216 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2858 reflections
θ = 2.3–28.3°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, yellow
0.48 × 0.28 × 0.24 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.972, T_{max} = 0.980
6663 measured reflections
2584 independent reflections
1929 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.5°
$h = -8 \rightarrow 8$
$k = -13 \rightarrow 9$
$l = -20 \rightarrow 20$

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Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.146$
$S = 1.08$
2584 reflections
167 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.3411P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.334 (2)	N2—C1	1.407 (2)
N1—C5	1.345 (2)	C6—O1	1.418 (3)
N2—C8	1.362 (2)	O2—C8	1.211 (2)
C1—N1—C5	118.45 (15)	C11—C9—C12	109.9 (2)
C8—N2—C1	129.17 (16)	C11—C9—C10	109.9 (2)
O2—C8—N2	122.54 (17)	C12—C9—C10	109.08 (18)
N2—C8—C9	115.01 (16)	C11—C9—C8	108.82 (16)
C8—N2—C1—C2	-7.1 (3)	O1—C6—C5—C4	112.2 (2)
N2—C1—C2—C3	178.94 (18)	C1—N2—C8—O2	4.6 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1$^{\text{i}}$	0.89 (3)	2.07 (3)	2.855 (2)	146 (3)
N2—H2*A*···O1$^{\text{i}}$	0.84 (2)	2.22 (2)	3.007 (2)	157 (2)
C2—H2···O2$^{\text{ii}}$	0.92 (2)	2.27 (2)	2.862 (2)	122 (2)

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

Methyl H atoms were placed in calculated positions and constrained to ride on their carrier atoms [C—H = 0.96 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C)]. The remaining H atoms were located in a difference map and refined isotropically. For refined H atoms, O—H = 0.89 (3) Å, N—H = 0.84 (2) Å and C—H = 0.92 (2)–1.00 (2) Å.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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