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1-[4-(Hexyloxy)phenyl]pyridin-4(1*H*)-one monohydrate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.007 Å
R factor = 0.071
wR factor = 0.280
Data-to-parameter ratio = 14.9

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

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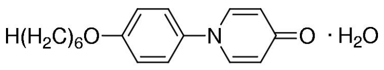
Structure analysis of the title compound, $C_{17}H_{21}NO_2\cdot H_2O$, containing a polar 4-pyridone moiety, demonstrates that bifurcated acceptor hydrogen bonds involving water molecules as hydrogen-bonding donors and C═O groups as hydrogen-bonding acceptors form infinite one-dimensional molecular chains.

Comment

Molecules with a pyridin-4(1*H*)-one (hereinafter referred to as 4-pyridone) unit have large birefringences (Dyer *et al.*, 1997), high anisotropic polarizabilities (Dirk *et al.*, 1986) and large dipole moments (Batts & Madeley, 1972). The rigid conjugated core of the 4-pyridone unit, reminiscent of the classical nCB and nOCB liquid crystal families (Gray, 1998), is also known to form strong intermolecular hydrogen-bonded (HB) interactions with HB donors (Hine & Ahn, 1987). Therefore, we are interested in incorporating the 4-pyridone unit into non-centrosymmetric hydrogen-bonded polymers (Landorf *et al.*, 2002). We use 1-[4-(hexyloxy)phenyl]pyridin-4(1*H*)-one (hereinafter referred to as C6-pyridone) as a model compound for studying the intermolecular HB interactions in 4-pyridone-containing HB polymers.



(I)

During synthesis of C6-pyridone, the elemental analysis data did not match the theoretical percentages for pure C6-pyridone; however, they did match the calculated percentage values for C6-pyridone monohydrate. X-ray crystal structure analysis confirmed that C6-pyridone crystallizes with water in a 1:1 molar ratio to form the title compound, (I).

The molecular structure of (I) is shown in Fig. 1 and some important bond lengths are given in Table 1. The 4-pyridone

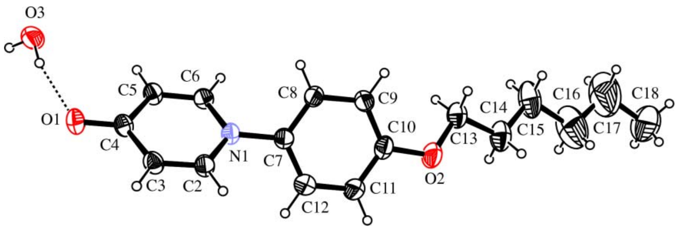


Figure 1
The molecular structure and atom-numbering scheme for (I), with displacement ellipsoids drawn at the 30% probability level. The dashed line indicates a hydrogen bond.

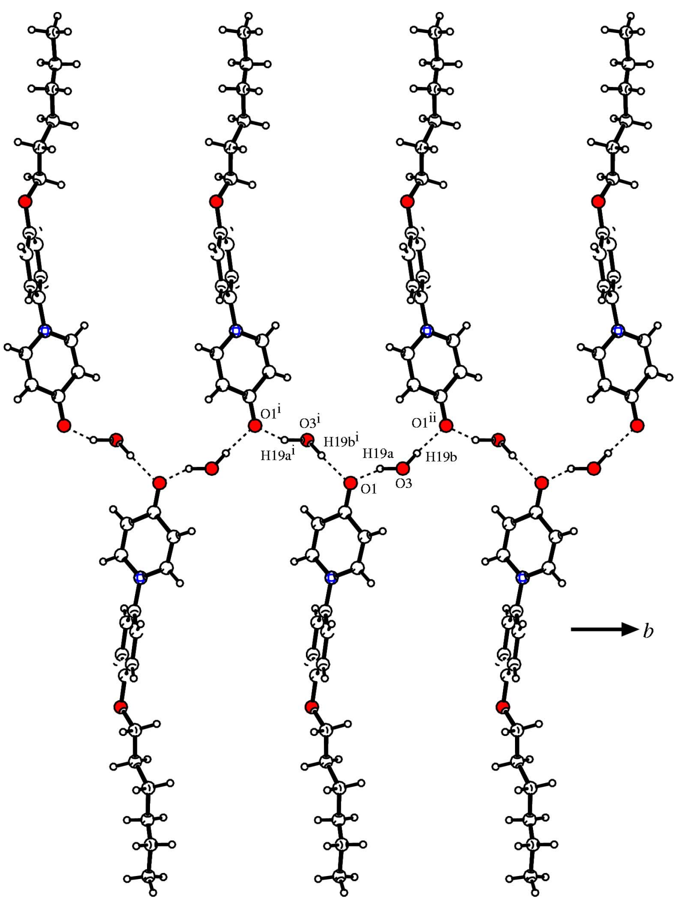


Figure 2
Hydrogen bonding (dashed lines) in (I). The bifurcated O—H···O(=C) bonds form infinite molecular chains parallel to the *b* axis. O atoms are colored red [symmetry codes: (i) $2 - x, y - \frac{1}{2}, \frac{5}{2} - z$; (ii) $2 - x, \frac{1}{2} + y, \frac{5}{2} - z$].

ring is planar within standard uncertainty. Carbonyl atom O1 is nearly coplanar with the 4-pyridone ring, deviating from the ring plane by only 0.021 (3) Å. The benzene ring is slightly less planar, the maximum deviation from its least-squares plane being 0.016 (4) Å (for atom C7). The dihedral angle between the two least-squares planes is 46.19 (19)°, slightly smaller than the corresponding angle of 50.39 (9)° in 1-(4-decylphenyl)pyridin-4(1*H*)-one (Li *et al.*, 2005). The alkoxyl tail with an 'all-*trans*' rigid conformation in the aliphatic chain, is twisted away from the benzene ring with a torsion angle of −23.9 (6)° (C13—O2—C10—C9), even though atom O2 is out of the benzene ring plane by only 0.031 (3) Å. In the polymorph structure, (III), of 4-octyloxybiphenyl-4′-carbonitrile (8OCB; Davey *et al.*, 2005), the dihedral angle between the biphenyl rings is 42°, with the alkoxyl tail almost coplanar with the attached benzene ring. Similarities in the aromatic ring dihedral angles between C6-pyridone and 8OCB suggest that the 4-pyridone core may induce liquid crystalline phases like the biphenyl core in 8OCB.

An analysis of the HB network shows that bifurcated acceptor O—H···O HB interactions form infinite polymeric chains along the *b* axis, as shown in Fig. 2. The C=O O atom acts as a hydrogen-bond acceptor to interact with two H atoms donated by two neighboring H_2O molecules, thus forming the bifurcated acceptor hydrogen bonds (Desiraju & Steiner, 1999). Details of the HB geometry are given in Table 2. The HB bond lengths and angles are all in the normal range of O_W—H···O(=C) HB interactions, with a mean $O_W\cdots A$ distance of 2.840 (2) Å (Steiner, 2002). However, the bifurcated acceptor HB interactions are not equivalent, as evidenced by the difference in the two *D*—*A* distances (Table 2). Such bifurcated acceptor hydrogen-bond interactions are commonly observed in N—H···O(=C) HB interactions in peptides and ureas, and are widely used in crystal engineering and supramolecular chemistry (Kishikawa *et al.*, 2005; Nguyen *et al.*, 2001); nevertheless, theoretical studies on their nature have not been thoroughly carried out (Yang & Gellman, 1998). The molecular packing is shown in Fig. 3. *PLATON* (Spek, 2003) calculations indicate no π stacking or π(arene) interactions.

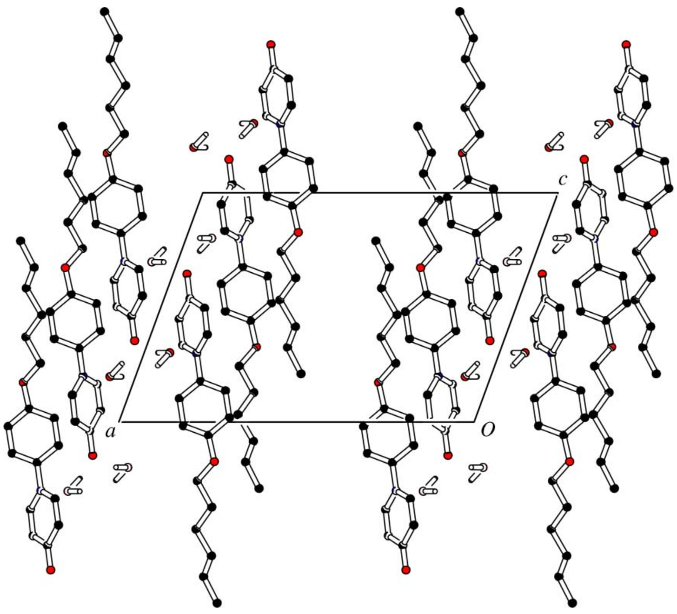


Figure 3
The molecular packing in (I), as viewed down the *b* axis. Most of the H atoms have been omitted to improve clarity.

Experimental

Compound (I) was synthesized according to the procedure described by You *et al.* (1999). Single crystals of (I) were obtained by slow room-temperature evaporation of a dilute toluene solution: m.p. 355.6 K (accompanied by release of H_2O). R_F = 0.41 (95:5, CH_2Cl_2/MeOH); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.56 (*d*, *J* = 6.9 Hz, 2H), 7.40 (*d*, *J* = 8.7 Hz, 2H), 7.12 (*d*, *J* = 8.7 Hz, 2H), 6.55 (*d*, *J* = 6.6 Hz, 2H), 3.97 (*s*, 2H), 1.82 (*m*, 2H), 1.32–1.50 (*m*, 6H), 0.89 (*t*, *J* = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 178.86, 159.15, 139.55, 136.23, 124.23, 118.75, 115.64, 68.53, 31.50, 29.05, 25.63, 22.56, 13.96. Analysis calculated for $C_{17}H_{23}NO_3$ (monohydrate): C 70.56, H 8.01, N 4.84%; found: C 70.61, H 8.06, N 4.73%. MS (EI, 70 eV) *m*/*z* (M^+): calculated for $C_{17}H_{21}NO_2$: 271.16; found: 271.2 (187.1, 159.1, 62.0).

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Crystal data

$C_{17}H_{21}NO_2 \cdot H_2O$
$M_r = 289.36$
Monoclinic, $P2_1/c$
$a = 17.815\ (5)$ Å
$b = 8.022\ (3)$ Å
$c = 12.251\ (3)$ Å
$\beta = 110.09\ (2)°$
$V = 1644.3\ (9)$ Å^3
$Z = 4$

$D_x = 1.169$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.0–11.0°$
$\mu = 0.08$ mm^{-1}
$T = 296$ K
Plate, colorless
$0.49 \times 0.41 \times 0.16$ mm

Data collection

Rigaku AFC-5*S* diffractometer
ω scans
Absorption correction: none
3075 measured reflections
2923 independent reflections
1400 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.017$

$\theta_{\rm max} = 25.0°$
$h = -21 \rightarrow 19$
$k = -9 \rightarrow 0$
$l = 0 \rightarrow 14$
3 standard reflections every 100 reflections intensity decay: 0.6%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.280$
$S = 1.04$
2923 reflections
196 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1703P)^2 + 0.0959P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.52$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.55$ e Å^{-3}

Table 1
Selected interatomic distances (Å).

O1—C4	1.263 (4)	N1—C7	1.439 (5)
N1—C2	1.367 (5)	C7—C12	1.377 (5)
N1—C6	1.360 (5)	C7—C8	1.376 (5)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H19*A*···O1	0.84 (5)	2.05 (5)	2.873 (5)	165 (5)
O3—H19*B*···O1$^{\rm i}$	0.82 (5)	2.03 (5)	2.847 (4)	177 (7)

Symmetry code: (i) $-x + 2, y + \frac{1}{2}, -z + \frac{5}{2}$.

Moderate disorder in the last three atoms of the alkoxyl tail of molecule (I) required the use of distance and angle restraints to prevent unreasonable geometry. Anisotropic displacement parameter restraints proved unhelpful. In the light of the disorder and the less than optimum data quality, methyl H atoms were positioned geometrically. The water H-atom positions were taken from a difference Fourier synthesis and refined with O—H distance restraints of 0.82 Å and fixed $U_{\rm iso}$ values equal to 1.5 times $U_{\rm eq}$ of the parent O atom. All other H atoms were treated as riding, with C—H distances ranging from 0.93 to 0.97 Å and $U_{\rm iso}$(H) values equal to 1.5 (methyl H atoms) or 1.2 (all other H atoms) times $U_{\rm eq}$ of the parent atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1996); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *PROCESS* in *TEXSAN* (Molecular Structure Corporation, 1997); program(s) used to solve structure: *SIR92* (Burla *et al.*, 1989); program(s) used to refine structure: *LS* in *TEXSAN* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek 2003); software used to prepare material for publication: *TEXSAN*, *SHELXL97* and *PLATON*.

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