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Key indicators

Single-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.053
 wR factor = 0.170
Data-to-parameter ratio = 9.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

4-(Hexylamino)phenol

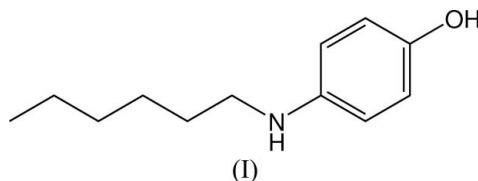
The title compound, $\text{C}_{12}\text{H}_{19}\text{NO}$, presents a two-dimensional hydrogen-bonding pattern, where amine and hydroxyl functionalities serve as both donors and acceptors.

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Comment

Secondary amines are common in polymer synthesis, where they are used as monomer chain extenders and crosslinking agents (Herman *et al.*, 1990; Choi *et al.*, 2004). The title compound, (I), is an amphiphilic monomer belonging to this class of compounds, containing a hydrophobic alkyl-chain moiety and a polar hydroxyl functionality. It was obtained by reacting 4-aminophenol with 1-bromohexane (see *Experimental*), demonstrating that, as expected, the amine functionality is more reactive than the hydroxy group when 4-aminophenol undergoes an $\text{S}_{\text{N}}2$ reaction with primary alkyl halides.The asymmetric unit of (I) consists of one molecule in a general position with the expected geometry (Fig. 1 and Table 1). O—H and N—H bonds are involved in a network of hydrogen bonds of moderate strength (Table 2); hydroxyl and amine functionalities serve both as donor and acceptor groups, forming a directed two-membered chain $\cdots\text{O}-\text{H}\cdots\text{N}-\text{H}\cdots\text{O}-\text{H}\cdots$ running along the short axis [100]. The complete network has a two-dimensional pattern (Fig. 2), giving an arrangement which avoids $\pi-\pi$ interactions; the dihedral angle between two adjacent phenol rings belonging to two hydrogen-bonded molecules is $78.99(9)^\circ$ (symmetry code: $1 - x, \frac{1}{2} + y, 1 - z$). The crystal structure is based on segregated stacks of aryl groups and alkyl groups, parallel to the [100] axis (Fig. 2, inset).

Experimental

To a solution of 4-aminophenol (1 g, 9 mmol) in cyclohexanone was added finely powdered K_2CO_3 (2.53 g, 18 mmol) under an argon atmosphere. The mixture was refluxed for 2 h and then 1-bromohexane (1.82 g, 11 mmol) was added in small portions. The reaction was continued under reflux for 24 h and the mixture was then cooled and filtered to remove KBr and unreacted K_2CO_3 . After evaporation of the solvent, the residue was dissolved in acetone and this solution was poured into hexane in order to precipitate unreacted 4-amino-

phenol and the solution reduced under reduced pressure at $T < 323$ K. This procedure was repeated until pure (I) was isolated as brown crystals (yield 0.82 g, 70%, m.p. 348 K). Analysis found: C 74.6, H 9.7, O 8.2, N 7.3%; calculated for $C_{12}H_{19}NO$: C 74.6, H 9.8, O 8.2, N 7.2%. 1H NMR (400 MHz, $CDCl_3$): δ 0.89 (m , $J = 8.8$ Hz, 3H, CH_3), 1.32 (m , $J = 7.2$, 6H, $3 \times CH_2$), 1.58 (m , $J = 6.8$ Hz, 2H, CH_2), 3.05 (m , $J = 6.8$ Hz, 2H, NCH_2), 4.01 (s , 1H, OH), 6.55 (d , $J = 8.4$ Hz, 2H, Ph), 6.68 (d , $J = 8.4$ Hz, 2H, Ph).

Crystal data

$C_{12}H_{19}NO$	Mo $K\alpha$ radiation
$M_r = 193.28$	Cell parameters from 55 reflections
Orthorhombic, $P2_12_12_1$	$\theta = 4.8$ – 11.9°
$a = 4.7016$ (6) Å	$\mu = 0.07$ mm $^{-1}$
$b = 10.1325$ (14) Å	$T = 296$ (1) K
$c = 26.273$ (3) Å	Prism, brown
$V = 1251.6$ (3) Å 3	0.55 \times 0.28 \times 0.20 mm
$Z = 4$	
$D_x = 1.026$ Mg m $^{-3}$	

Data collection

Bruker P4 diffractometer	$\theta_{max} = 25.0^\circ$
$2\theta/\omega$ scans	$h = -5 \rightarrow 5$
Absorption correction: none	$k = -12 \rightarrow 12$
5524 measured reflections	$l = -31 \rightarrow 31$
1327 independent reflections	3 standard reflections
833 reflections with $I > 2\sigma(I)$	every 97 reflections
$R_{int} = 0.048$	intensity decay: 3%

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.053$	$w = 1/[\sigma^2(F_o^2) + (0.1)^2]$
$wR(F^2) = 0.170$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
1327 reflections	$\Delta\rho_{max} = 0.11$ e Å $^{-3}$
136 parameters	$\Delta\rho_{min} = -0.17$ e Å $^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1–C1	1.377 (4)	C7–C8	1.527 (5)
O1–H1A	0.72 (5)	C8–C9	1.486 (6)
N1–C4	1.419 (4)	C9–C10	1.549 (7)
N1–C7	1.449 (5)	C10–C11	1.466 (8)
N1–H1B	0.75 (5)	C11–C12	1.494 (8)
C1–O1–H1A	114 (3)	C4–N1–H1B	107 (3)
C4–N1–C7	118.6 (3)	C7–N1–H1B	107 (3)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1–H1A \cdots N1 ⁱ	0.72 (5)	2.08 (5)	2.764 (5)	158 (5)
N1–H1B \cdots O1 ⁱⁱ	0.75 (5)	2.34 (5)	3.058 (5)	161 (4)

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

The H atoms bonded to heteroatoms N1 and O1 were found in a difference map and refined with free coordinates and isotropic U parameters. H atoms bonded to C atoms were placed in idealized positions and refined as riding on their parent C atom [C–H

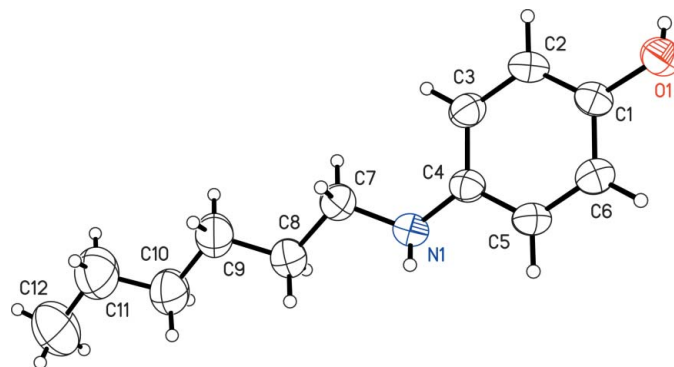


Figure 1

The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

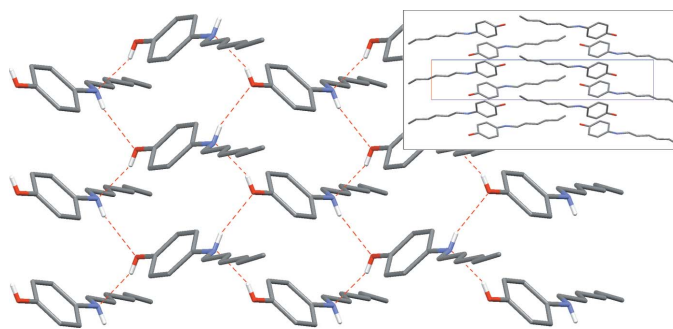


Figure 2

Part of the crystal structure of (I), showing the hydrogen-bonding scheme (dashed red lines). For clarity, H atoms bonded to C atoms have been omitted. The inset is a projection of the crystal structure along [010]. The short cell axis corresponds to the a axis and the long axis corresponds to the c axis.

distances and isotropic $U_{iso}(H)$ parameters: methylene 0.97 Å and $1.2U_{eq}(C)$; methyl 0.96 Å and $1.5U_{eq}(C)$; aromatic 0.93 Å and $1.2U_{eq}(C)$). In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL-Plus*; molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXTL-Plus*.

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