Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 4-[(3-Pyridylamino)methyl]phenol

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.058
*wR* factor = 0.133
Data-to-parameter ratio = 16.0

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

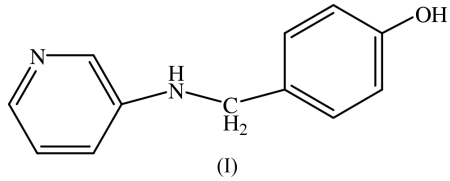


Received 20 March 2001
Accepted 21 March 2001
Online 12 April 2001

The title compound, $C_{12}H_{12}N_2O$, crystallizes in the non-centrosymmetric $P2_12_12_1$ space group giving crystals showing a second harmonic generation (SHG) property. The crystal structure consists of discrete molecules and forms a three-dimensional network through intermolecular hydrogen bonding.

## Comment

The title compound, (I), is shown in Fig. 1. These molecules are packed in a non-centrosymmetric structure, probably as a consequence of the two hydrogen-bonding interactions that are found involving the phenol hydroxyl group, which acts as both donor and acceptor, the amino group acting as donor, and the pyridine N atom acting as acceptor. O—H···N hydrogen bonds join the molecules 'head-to-tail' in chains running along the [001] direction with a *C*(11) motif (Etter *et al.*, 1990), while N—H···O bonds join the chains along [010] with a *C*(8) motif, the combination of the two chains resulting in sheets (see Fig. 2).




(I)

All bond lengths and angles are in the normal ranges. The dihedral angle between the two aromatic ring planes is 63.4 (2)°. The conformation along the C8—N1—N7—C6 central chain is given by the torsion angles C9—C8—N1—N7 of 171.6 (3), C8—N1—C7—C6 of 174.1 (2) and N1—C7—C6—C5 of 128.7 (3)°.

## Experimental

4-Hydroxybenzaldehyde (6.15 g, 50 mmol) and 3-aminopyridine (4.71 g, 50 mmol) in 100 ml of toluene were refluxed with a Dean–Stark trap for 12 h. Toluene was removed *in vacuo*, and the residue was dissolved in 100 ml of ethanol. $NaBH_4$ (3.70 g, 100 mmol) was added to the ethanol solution and the resultant mixture was stirred at room temperature for 18 h. Excess $NaBH_4$ was quenched with water and then with saturated $NH_4Cl$ solution at 343 K. A pale-yellow solid powder was obtained through filtration (yield: 7.20 g, 72.0%). Crystals suitable for single-crystal X-ray diffraction studies were obtained by hydrothermal treatment of $W(CO)_6$ and 4-[(3-pyridinylamino)-methyl]phenol in methanol at 353 K. The structure of the title compound was confirmed by IR analysis [3338 (*s*), 3021 (*w*), 2804 (*w*),

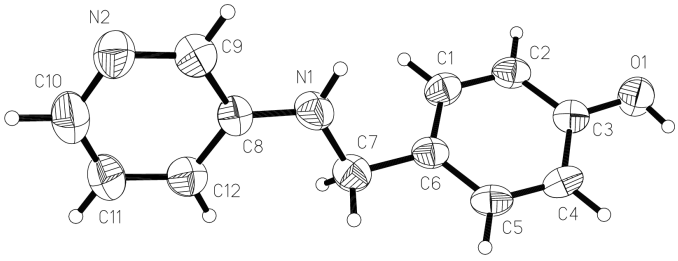



**Figure 1**
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

2680 (*w*), 1597 (*s*), 1576 (*s*), 1512 (*s*), 1463 (*m*), 1341 (*m*), 1320 (*m*), 1276 (*s*), 828 (*m*), 790 (*m*) and 698 (*w*)]. Tests on the powdered title compound show a positive signal for SHG.

*Crystal data*

$C_{12}H_{12}N_2O$
$M_r$ = 200.24
Orthorhombic, $P2_12_12_1$
$a$ = 6.5392 (2) Å
$b$ = 7.6077 (1) Å
$c$ = 20.3127 (5) Å
$V$ = 1010.52 (4) Å$^3$
$Z$ = 4
$D_x$ = 1.316 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 2653 reflections
$\theta$ = 2.0–29.5°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 (2) K
Rectangular block, yellow
0.20 × 0.14 × 0.01 mm

*Data collection*

Siemens SMART CCD area-detector diffractometer
$\omega$ scans
6968 measured reflections
2308 independent reflections
1400 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.079
$\theta_{max}$ = 27.5°
$h = -5 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -26 \rightarrow 24$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.133
$S$ = 0.99
2308 reflections
144 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O1—C3 | 1.363 (3) | N2—C9 | 1.322 (3) |
| N1—C8 | 1.368 (3) | N2—C10 | 1.351 (4) |
| N1—C7 | 1.453 (3) | | |
| C3—O1—H1*A* | 113 (2) | C7—N1—H1*B* | 112 (2) |
| C8—N1—C7 | 120.3 (2) | C9—N2—C10 | 117.5 (3) |
| C8—N1—H1*B* | 120 (2) | | |

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1*A*···N2$^{i}$ | 0.98 (4) | 1.75 (4) | 2.723 (3) | 170 (4) |
| N1—H1*B*···O1$^{ii}$ | 0.89 (3) | 2.26 (3) | 3.133 (3) | 166 (3) |

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

After checking their presence in the difference map, all H atoms were geometrically fixed and allowed to ride on their attached atoms, except for the H atoms involved in hydrogen bonding which were refined isotropically. It was not possible to define the correct absolute configuration as all the atoms were too weak anomalous scatterers of Mo $K\alpha$ radiation.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and SADABS (Sheldrick,

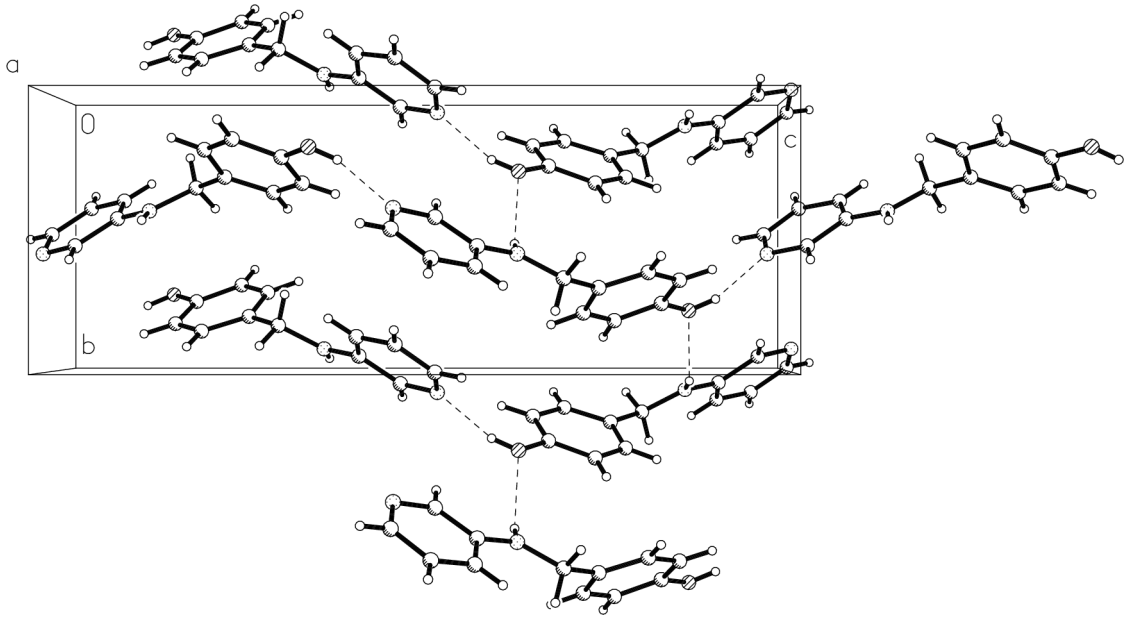



**Figure 2**
Packing diagram of (I) viewed down the *a* axis. O—H···N and N—H···O hydrogen-bond contacts are shown as dashed lines.

1996); 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, 1990).

This work was funded by The Major State Basic Research Development Program (grant No. G2000077500) and the National Natural Science Foundation of China. The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 305/PFIZIK/610942.

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