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2-Bromo-3-hydroxy-6-methylpyridine

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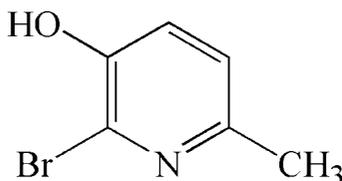
Received 14 October 2013; accepted 25 October 2013

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.025; wR factor = 0.066; data-to-parameter ratio = 15.3.

In the title compound, $\text{C}_6\text{H}_6\text{BrNO}$, the Br atom is displaced from the pyridine ring mean plane by 0.0948 (3) Å, while the hydroxyl O atom and the methyl C atom are displaced by 0.0173 (19) and 0.015 (3) Å, respectively. In the crystal, molecules are linked *via* $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds, forming chains propagating along the a -axis direction. These chains are linked by $\text{C}-\text{H}\cdots\text{Br}$ hydrogen bonds, forming corrugated two-dimensional networks lying parallel to the ac plane.

Related literature

3-Hydroxypyridine, the core skeleton of the title compound is an integral part of Nikkomycin Z (a potent fungicide), see: Tetsu *et al.* (1990). For the synthesis, see: Kjell *et al.* (1969).



Experimental

Crystal data

 $\text{C}_6\text{H}_6\text{BrNO}$
 $M_r = 188.03$

 Orthorhombic, $Pbca$
 $a = 11.4484$ (19) Å

 $b = 9.0914$ (15) Å

 $c = 13.230$ (2) Å

 $V = 1377.1$ (4) Å³
 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 5.88$ mm⁻¹
 $T = 298$ K

 $0.32 \times 0.22 \times 0.12$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

 Absorption correction: multi-scan (*SADABS*; Bruker, 2001)

 $T_{\min} = 0.255$, $T_{\max} = 0.539$

12822 measured reflections

1335 independent reflections

 1115 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.066$
 $S = 1.06$

1335 reflections

87 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.36$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}^i$	0.80 (3)	1.92 (3)	2.717 (3)	174 (3)
$\text{C6}-\text{H6B}\cdots\text{Br1}^{ii}$	0.96	3.04	3.993 (3)	174

 Symmetry codes: (i) $x + \frac{1}{2}, y, -z + \frac{3}{2}$; (ii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *X-SEED* (Barbour, 2001).

The authors thank Bhagavan Sri Sathya Sai Baba for constant guidance and motivation. We would like to thank Professor Ashwini Nangia, University of Hyderabad, for his help with the single-crystal X-ray diffraction facility. GNR acknowledges financial support from the Council of Scientific and Industrial Research (CSIR), 01 (2286)/08/EMR-II, India. NRG thanks the CSIR for a fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2659).

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supporting information

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2-Bromo-3-hydroxy-6-methylpyridine

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S1. Comment

3-Hydroxypyridine is an integral part of Nikkomycin Z (NZ), a potent fungicide, insecticide, miticide, and inhibitor of fungal and insect chitin synthetase (Tetsu *et al.*, 1990). Various biaryl derivative compounds, derived originally from 3-hydroxypyridine, are PDE4 inhibitors useful for the treatment and prevention of strokes, myocardial infarction and cardiovascular inflammatory diseases and disorders. Herein we describe the crystal structure of the 2-bromo derivative of 3-hydroxy-6-methylpyridine, previously synthesized by (Kjell *et al.*, 1969).

The molecular structure of the title molecule is illustrated in Fig. 1. The bond lengths and angles are normal.

In the crystal, molecules are linked via O-H \cdots N hydrogen bonds forming chains propagating along the a axis direction (Fig. 2 and Table 1). These chains are linked by weak C-H \cdots Br hydrogen bonds forming corrugated two-dimensional networks lying parallel to the ac plane (Fig. 2 and Table 1).

S2. Experimental

The title compound was synthesized following the published procedure (Kjell *et al.*, 1969). Colourless crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a solution of the title compound in ethanol [m.p. = 460–462 K].

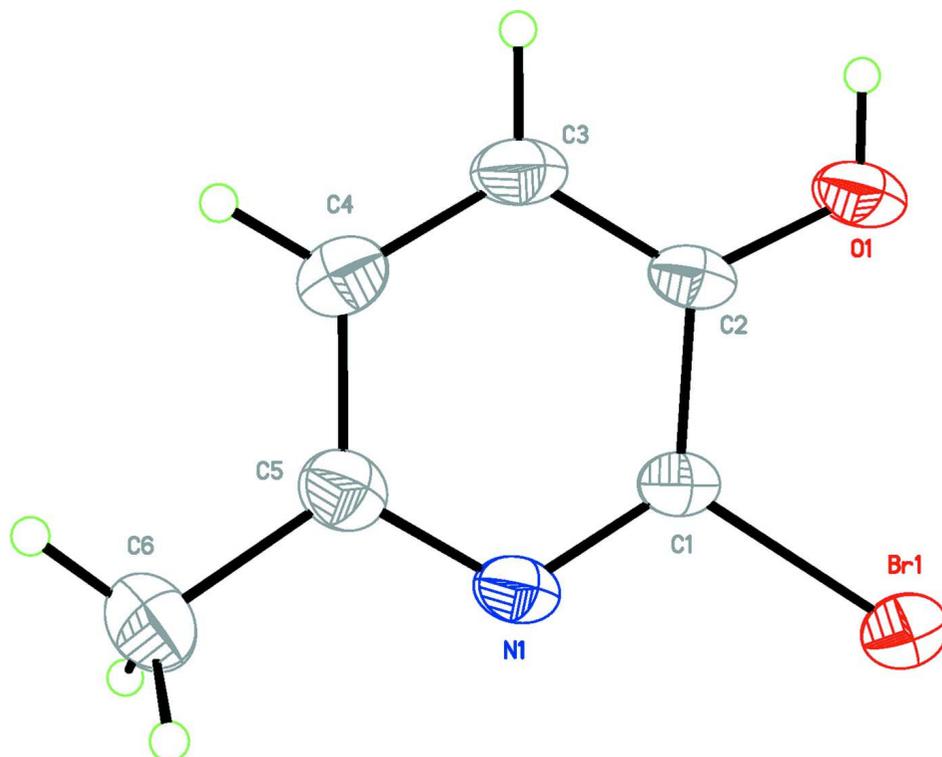


Figure 1

A view of the molecular structure of the title molecule, with atom labelling. Displacement ellipsoids are drawn at the 35% probability level.

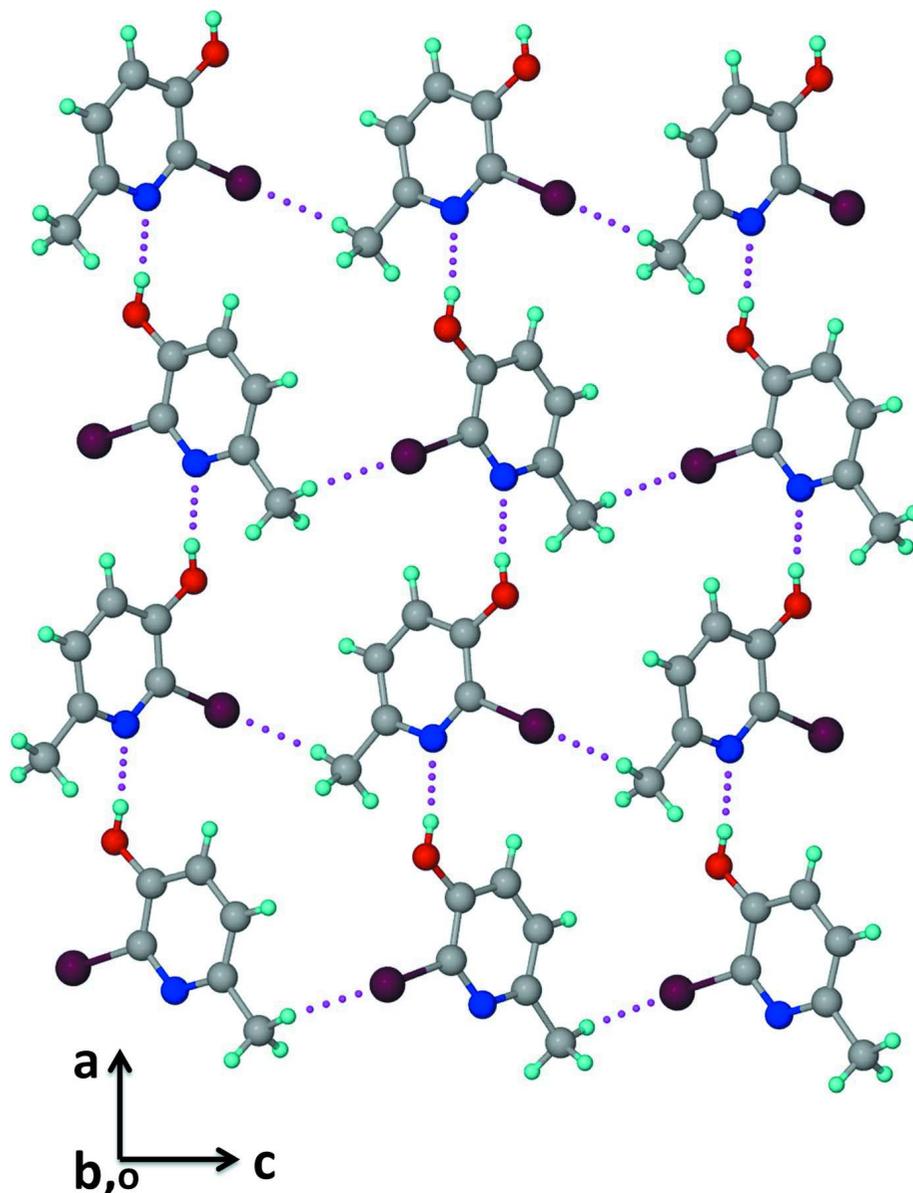


Figure 2

A view normal to the *ac* plane of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1 for details; *a* axis vertical; *c* axis horizontal).

2-Bromo-3-hydroxy-6-methylpyridine

Crystal data

C_6H_6BrNO

$M_r = 188.03$

Orthorhombic, *Pbca*

Hall symbol: $-P\ 2ac\ 2ab$

$a = 11.4484\ (19)\ \text{\AA}$

$b = 9.0914\ (15)\ \text{\AA}$

$c = 13.230\ (2)\ \text{\AA}$

$V = 1377.1\ (4)\ \text{\AA}^3$

$Z = 8$

$F(000) = 736$

$D_x = 1.814\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 4373 reflections

$\theta = 3.1\text{--}25.7^\circ$

$\mu = 5.88\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Needle, colorless

$0.32 \times 0.22 \times 0.12\ \text{mm}$

Data collection

Bruker SMART CCD area-detector
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2001)
 $T_{\min} = 0.255$, $T_{\max} = 0.539$

12822 measured reflections
1335 independent reflections
1115 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$
 $\theta_{\max} = 25.9^\circ$, $\theta_{\min} = 3.1^\circ$
 $h = -14 \rightarrow 14$
 $k = -11 \rightarrow 11$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.066$
 $S = 1.06$
1335 reflections
87 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0376P)^2 + 0.3754P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.36 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.71386 (2)	1.06988 (3)	0.63569 (2)	0.0542 (1)
O1	0.95756 (15)	0.9896 (2)	0.69349 (15)	0.0603 (7)
N1	0.68110 (16)	0.8898 (2)	0.79970 (15)	0.0419 (6)
C1	0.76362 (18)	0.9477 (2)	0.74339 (17)	0.0380 (6)
C2	0.88204 (18)	0.9217 (2)	0.75492 (19)	0.0422 (7)
C3	0.9120 (2)	0.8254 (3)	0.83193 (19)	0.0498 (8)
C4	0.8269 (2)	0.7633 (3)	0.89095 (18)	0.0506 (8)
C5	0.7110 (2)	0.7963 (3)	0.87433 (17)	0.0466 (8)
C6	0.6136 (2)	0.7345 (4)	0.9362 (2)	0.0664 (10)
H1	1.023 (3)	0.961 (3)	0.700 (2)	0.075 (10)*
H3	0.99010	0.80290	0.84360	0.0600*
H4	0.84740	0.69860	0.94240	0.0610*
H6A	0.57840	0.81170	0.97520	0.1000*
H6B	0.64370	0.66040	0.98090	0.1000*
H6C	0.55610	0.69160	0.89240	0.1000*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0429 (2)	0.0644 (2)	0.0552 (2)	0.0084 (1)	-0.0049 (1)	0.0091 (1)
O1	0.0266 (9)	0.0798 (13)	0.0746 (13)	0.0023 (8)	0.0044 (8)	0.0172 (11)
N1	0.0281 (8)	0.0498 (10)	0.0479 (11)	-0.0018 (8)	-0.0013 (8)	-0.0035 (9)
C1	0.0286 (10)	0.0420 (12)	0.0434 (11)	0.0032 (9)	-0.0032 (9)	-0.0038 (9)
C2	0.0252 (10)	0.0493 (13)	0.0520 (13)	-0.0004 (9)	-0.0013 (9)	-0.0039 (10)
C3	0.0304 (11)	0.0610 (15)	0.0580 (14)	0.0062 (11)	-0.0076 (10)	-0.0011 (12)
C4	0.0447 (13)	0.0576 (15)	0.0496 (13)	0.0047 (12)	-0.0095 (10)	0.0038 (12)
C5	0.0395 (13)	0.0516 (14)	0.0486 (14)	-0.0036 (10)	0.0007 (10)	-0.0032 (10)
C6	0.0535 (15)	0.0822 (19)	0.0634 (17)	-0.0139 (14)	0.0056 (13)	0.0121 (15)

Geometric parameters (\AA , $^\circ$)

Br1—C1	1.894 (2)	C4—C5	1.378 (3)
O1—C2	1.338 (3)	C5—C6	1.493 (4)
O1—H1	0.80 (3)	C3—H3	0.9300
N1—C5	1.347 (3)	C4—H4	0.9300
N1—C1	1.313 (3)	C6—H6A	0.9600
C1—C2	1.385 (3)	C6—H6B	0.9600
C2—C3	1.386 (3)	C6—H6C	0.9600
C3—C4	1.370 (3)		
C2—O1—H1	113 (2)	N1—C5—C4	119.9 (2)
C1—N1—C5	119.07 (19)	C2—C3—H3	120.00
Br1—C1—N1	116.45 (15)	C4—C3—H3	120.00
N1—C1—C2	125.0 (2)	C3—C4—H4	120.00
Br1—C1—C2	118.52 (16)	C5—C4—H4	120.00
O1—C2—C1	119.2 (2)	C5—C6—H6A	109.00
C1—C2—C3	115.5 (2)	C5—C6—H6B	109.00
O1—C2—C3	125.3 (2)	C5—C6—H6C	110.00
C2—C3—C4	120.2 (2)	H6A—C6—H6B	109.00
C3—C4—C5	120.3 (2)	H6A—C6—H6C	109.00
N1—C5—C6	116.7 (2)	H6B—C6—H6C	109.00
C4—C5—C6	123.4 (2)		
C5—N1—C1—Br1	176.80 (17)	N1—C1—C2—O1	-178.9 (2)
C5—N1—C1—C2	-0.8 (3)	C1—C2—C3—C4	-0.2 (3)
C1—N1—C5—C6	179.7 (2)	O1—C2—C3—C4	179.3 (2)
C1—N1—C5—C4	0.3 (3)	C2—C3—C4—C5	-0.1 (4)
Br1—C1—C2—C3	-176.81 (17)	C3—C4—C5—C6	-179.2 (3)
Br1—C1—C2—O1	3.6 (3)	C3—C4—C5—N1	0.1 (4)
N1—C1—C2—C3	0.7 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots N1 ⁱ	0.80 (3)	1.92 (3)	2.717 (3)	174 (3)
C6—H6B \cdots Br1 ⁱⁱ	0.96	3.04	3.993 (3)	174

Symmetry codes: (i) $x+1/2, y, -z+3/2$; (ii) $x, -y+3/2, z+1/2$.