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Structure of 4-Bromo-2-(2-pyridyliminomethyl)phenol

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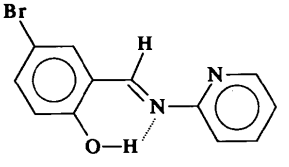
Abstract. (1) $C_{12}H_9BrN_2O$, $M_r = 277{\cdot}3$, monoclinic, $P2_1/n$, $a = 8{\cdot}167$ (3), $b = 17{\cdot}541$ (5), $c = 8{\cdot}457$ (3) Å, $\beta = 114{\cdot}67$ (3)°, $V = 1100$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}67$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}367$ mm^{-1}, $F(000) = 552$, room temperature, $R = 0{\cdot}041$ for 1194 unique observed reflections. The exocyclic C and the pyridine N atoms are in *cis* positions. A strong intramolecular O—H···N bond [O···N = 2·619 (8) Å] leads to the formation of a supplementary ring. The molecule is not planar; the pyridine ring is inclined at 5·20° to the phenyl ring, and at 6·1° to the hydrogen-bonded ring.

Experimental. Compound (1) was prepared in methanol by condensation of 5-bromosalicylaldehyde with 2-aminopyridine in equimolar ratio.



(1)

When the reaction mixture cooled, orange crystals were obtained. Crystal dimensions 0·10 × 0·09 × 0·30 mm, Enraf–Nonius CAD-4 diffractometer, cell dimensions from 2θ of 25 reflections ($\theta < 11°$), $2\theta_{max} = 27°$, scan $\omega/2\theta = 1$, $t_{max} = 60$ s, range hkl 0,9; 0,20; −9,9, no appreciable decay of the intensity control reflections. The data reduction (Lorentz and polarization correction, no absorption correction) gives 1802 unique reflections after averaging ($R_{int} = 0{\cdot}017$) and 1194 with $I > 3\sigma(I)$. Structure solution by *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), refined by full-matrix least squares based on F, weights based on counting statistics; $1/w = \sigma^2(F) = 1/4[\sigma^2(I) + (0{\cdot}04I)^2]/I$ (Stout & Jensen, 1968); atomic scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV); H atoms from a difference Fourier map and their positions refined with fixed thermal parameters (4·0 Å^2); $R = 0{\cdot}041$ and $wR = 0{\cdot}042$; goodness of fit $S = 1{\cdot}37$ for refined parameters; largest shift over e.s.d. in the last cycle = 0·02; largest residual peak in final map = 0·26 e Å^{-3} (except peaks near Br atom). Calculations performed with *SDP-Plus* (Frenz, 1983) package and *XRAY*76 system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Table 1 lists the fractional coordinates, Table 2 bond lengths and bond angles;* Fig. 1 shows the molecular structure with the atomic numbering of the title compound.

Related literature. The present work continues the structural studies of Schiff bases. 4-Bromo-2-(2-pyridyliminomethyl)phenol (4-Br-salapy) (1) has been prepared and its structure determined in order to make a comparison with 2-(4-methyl- 2-pyridyliminomethyl)phenol (salampy) and 2-(4,6-dimethyl-2-pyridyliminomethyl)phenol (saladimpy) reported previously (Escobar & Garland, 1983,

* Lists of structure factors, anisotropic thermal parameters, torsion angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53696 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic positional parameters and B_{eq} values for* (1), *with e.s.d.'s*

$B_{eq} = (4/3)[a^2B_{11} + b^2B_{22} + c^2B_{33} + (2ab\cos\gamma)B_{12} + (2ac\cos\beta)B_{13} + (2bc\cos\alpha)B_{23}]$.

	x	y	z	B_{eq}(Å^2)
Br	0·28344 (7)	0·11981 (3)	0·07820 (6)	4·15 (1)
O	−0·2308 (5)	0·1133 (2)	−0·6789 (4)	4·58 (9)
N1	−0·3496 (5)	−0·0173 (2)	−0·6189 (5)	2·96 (9)
N2	−0·4606 (5)	−0·1322 (2)	−0·5533 (5)	3·5 (1)
C1	0·0105 (6)	0·0545 (3)	−0·2247 (5)	3·1 (1)
C2	0·1231 (6)	0·1160 (3)	−0·1620 (6)	3·1 (1)
C3	0·1201 (6)	0·1751 (3)	−0·2688 (7)	3·7 (1)
C4	0·0031 (6)	0·1739 (3)	−0·4422 (6)	3·7 (1)
C5	−0·1156 (6)	0·1123 (3)	−0·5095 (6)	3·2 (1)
C6	−0·1100 (6)	0·0524 (3)	−0·3981 (5)	2·7 (1)
C7	−0·2319 (6)	−0·0131 (3)	−0·4610 (5)	3·0 (1)
C8	−0·4661 (5)	−0·0817 (3)	−0·6724 (5)	2·9 (1)
C9	−0·5769 (6)	−0·0885 (3)	−0·8458 (6)	3·4 (1)
C10	−0·6871 (6)	−0·1520 (3)	−0·9028 (6)	3·7 (1)
C11	−0·6829 (6)	−0·2049 (3)	−0·7830 (7)	3·6 (1)
C12	−0·5697 (7)	−0·1925 (3)	−0·6117 (6)	3·9 (1)
H'O	−0·276 (5)	0·076 (3)	−0·697 (5)	4*

* Refined isotropically.

Table 2. *Selected bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

O—C5	1·346 (5)	C3—C4	1·376 (6)
O—H'O	0·73 (5)	C4—C5	1·404 (7)
N1—C7	1·281 (5)	C5—C6	1·399 (7)
N1—C8	1·424 (6)	C6—C7	1·466 (6)
N2—C8	1·329 (6)	C8—C9	1·369 (6)
N2—C12	1·337 (6)	C9—C10	1·386 (7)
C1—C2	1·371 (6)	C10—H10	0·98 (4)
C1—C6	1·384 (5)	C11—C12	1·372 (6)
C2—C3	1·369 (7)		

C5—O—H'O	106 (3)	C1—C6—C7	118·7 (4)
C7—N1—C8	119·2 (4)	C5—C6—C7	121·2 (3)
C8—N2—C12	116·1 (4)	N1—C7—C6	121·6 (4)
C2—C1—C6	119·8 (4)	N1—C8—N2	118·8 (3)
C1—C2—C3	121·0 (4)	N1—C8—C9	117·7 (4)
C2—C3—C4	120·3 (4)	N2—C8—C9	123·5 (4)
C3—C4—C5	119·9 (5)	C8—C9—C10	119·0 (5)
O—C5—C4	118·6 (4)	C9—C10—C11	118·5 (4)
O—C5—C6	122·6 (4)	C10—C11—C12	118·2 (5)
C4—C5—C6	118·8 (4)	N2—C12—C11	124·6 (5)
C1—C6—C5	120·1 (4)		

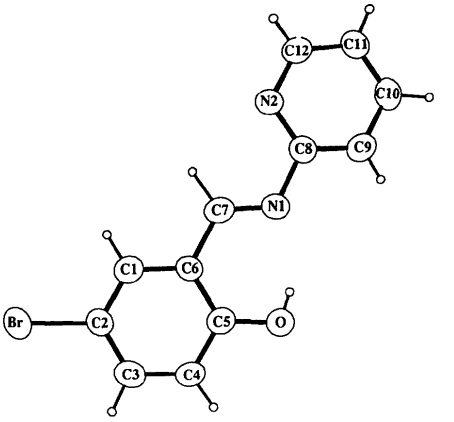

Fig. 1. Molecular structure of the title compound, with atom-numbering scheme.

1984). The 5-bromo derivative was synthetized because 2-(2-pyridyliminomethyl)phenol (salapy) did not give good crystals for X-ray diffraction studies.

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Structure of a Fully Protected Seco-erythronolide B Acid Derivative

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Abstract. (3*S*,4*R*,5*S*,6*R*,7*R*,9*R*,10*S*,11*S*,12*S*,13*S*,-14*R*)-14-Benzyloxymethoxy-10,12-*O*-carbonyl-4-*N*-imidazolylcarbonyl-6,7-*O*-isopropylidene-3,5,7,9,11,-13-hexamethylhexadec-1-ene-4,6,7,10,12,14-hexol, $C_{38}H_{56}N_2O_9$, $M_r = 684{\cdot}87$, monoclinic, $P2_1$, $a = 8{\cdot}7302$ (12), $b = 15{\cdot}597$ (2), $c = 14{\cdot}463$ (2) Å, $\beta = 104{\cdot}797$ (10)°, $V = 1904{\cdot}1$ (4) Å^3, $Z = 2$, D_x (198 K) $= 1{\cdot}19$ g cm^{-3}, $\mu = 0{\cdot}7893$ cm^{-1}, Mo $K\alpha$ radiation,

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