producing weaker (and longer) S—X bonds (where $X = $ C or O) and smaller X—S—X' bond angles (Day, Kingsbury & Day, 1981; Hoyos-Guerrero, Martínez-Carrera & García-Blanco, 1983). O(3) is twisted out of the plane of the phenyl ring. The pertinent torsion angles are O(3)—S—C(13)—C(14) = −6·3 (6) and O(3)-S—C(13)—C(18) = 167·6 (5)°.

The 1,3-dioxane ring adopts a chair conformation. The best *anti* relation between vicinal atoms is found with torsion angles O(1)—C(1)—C(6)—C(5) and C(3)—C(2)—S—C(13), 62·5 (6) and −80·9 (4)°. However, the angle between the normals to the planes of the 1,3-dioxane and phenyl rings is only 27·4 (5)°.

There is no intramolecular interaction between the phenyl ring and the methoxycarbonyl group; the planes defined by C(13)—C(18) and C(4), C(12), O(4), O(5), C(19) are nearly perpendicular [114·2 (5)°] (Fig. 1).

The 1,3-dioxane ring and the methoxycarbonyl group are oriented to minimize transannular repulsions between their O atoms and the sulfoxide group and cyclohexane ring [O(1)···S, 2·918 (4); O(2)···S, 3·026 (4); O(3)···O(1), 3·16 (1); and O(4)···C(2), 3·26 (1) Å], forming a spiral-like structure.

There are no intermolecular contacts short enough to affect the conformation of the molecule. The shortest intermolecular distances between non-H atoms are C(6)···O(3) (−0·5 + x, 1·5 − y, z) 3·38 (1) and

C(11)···S (−x, 2 − y, −z) 3·80 (1) Å. The packing in the crystal is entirely due to van der Waals forces.

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Structure of 2-(4,6-Dimethyl-2-pyridyliminomethyl)phenol, C₁₄H₁₄N₂O

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Abstract. $M_r = 226·3$, orthorhombic, $P2_12_12_1$, $a = 21·585$ (5), $b = 8·8285$ (8), $c = 6·2952$ (6) Å (JCPDS Diffraction File No. 33-1998), $V = 1199·6$ (3) Å³, $Z = 4$, $D_m = 1·22$ (3) (flotation in dioxane and bromoform), $D_x = 1·253$ (1) Mg m⁻³, $\lambda(\text{Mo } K\alpha_1) = 0·7093$ Å, $\mu = 0·09$ mm⁻¹, $F(000) = 480$, $T = 294$ K, final $R = 0·073$ for 1054 observed reflections. The molecule is not planar; the pyridine-ring plane is inclined at 5·4 (9)° to the salicylidene-ring plane. The orientation adopted by the pyridine ring is such that the exocyclic C atom is *cis* to the pyridine N. There is a strong intramolecular O—H···N hydrogen bond, O···N 2·60 (1) Å.

Introduction. The present work continues investigations on the structures of Schiff bases derived from salicylaldehyde and 2-aminopyridine.

Experimental. Dark yellow crystals (from methanol solution), 0·21 × 0·21 × 0·34 mm. Philips PW 1100 diffractometer, graphite-monochromatized Mo $K\alpha$, $\omega/2\theta$ scan mode. Two standard reflections every 55 measurements (variation ±2%). 2040 independent hkl with $2\theta < 60°$, 986 unobserved with $I < 2\sigma(I)$. Lp correction, absorption and anomalous dispersion ignored. 52 reflections with $2\theta < 47°$ used for refining cell parameters. Direct methods (*MULTAN*76, Main,

Lessinger, Woolfson, Germain & Declercq, 1976). Anisotropic full matrix. H from OH group from ΔF synthesis, others calculated, all refined with fixed isotropic B equal to the B_{eq} of the parent atoms. $\sum w\Delta^2$ $(\Delta = |F_o| - |F_c|)$ minimized; $w = 1$ if $\sin\theta > 0\cdot28$, otherwise $w = \sin\theta/0\cdot28$. $R = 0\cdot073$, $R_w = 0\cdot075$, $S = |\sum w\Delta^2/(m-n)|^{1/2} = 1\cdot87$ for the 1054 (m) reflections with $I > 2\sigma(I)$ used in the refinement of 196 (n) parameters; overdetermination ratio (m/n) is $5\cdot4$ and $R = \sum\Delta/\sum F = 0\cdot12$ for the 2040 unique reflections measured. $(\Delta/\sigma)_{max}$ for non-hydrogen atoms $= 0\cdot18$, for H atoms $0\cdot30$. $\Delta\rho$ residuals $-0\cdot34$ to $0\cdot23$ e Å^{-3}. Scattering factors for O,N,C (Cromer & Mann, 1968), for H (Stewart, Davidson & Simpson, 1965). Calculations carried out on an IBM 370/145 computer using the $XRAY76$ system (Stewart, 1976). Mean planes and torsion angles with $NRC22$ program (Ahmed, Hall, Pippy & Huber, 1973).

Discussion. The molecular structure with the thermal motion and the atomic numbering for the title compound (saladimpy) are shown in Fig. 1. The final positional and isotropic thermal parameters are listed in Table 1.*

In what follows a comparison is made with 2-(4-methyl-2-pyridyliminomethyl)phenol, $C_{13}H_{12}N_2O$ (salampy) (Escobar & Garland, 1983).

Both molecules have identical conformations with the hetero N atom of the pyridine ring cis to the exocyclic H(7). The intramolecular hydrogen bond between the imino atom and the O atom has distances N—O $= 2\cdot60$ (1), N—H $= 1\cdot97$ (6), O—H $= 0\cdot76$ (6) Å and interbond angle O—H$\cdots$N $= 140$ (6)°. The C—C aromatic bonds in the salicylidene ring range from $1\cdot37$ (1) to $1\cdot41$ (1) Å (Table 2). The smallest values correspond to the usual short C(3)—C(4), C(5)—C(6) bonds encountered in N-salicylidene-2-aminopyridine (Moustakali-Mavridis, Hadjoudis & Mavridis, 1978) and in N-salicylidene-3-aminopyridine (Moustakali-Mavridis, Hadjoudis & Mavridis, 1980). The short C—O bond of $1\cdot34$ (1) Å is normal in this type of compound. All these values are comparable to those of salampy. The exocyclic C—C bond is short $[1\cdot45$ (1) Å] in agreement with the partial double-bond character of a formal single bond between an aromatic and a double bond (Mavridis & Moustakali-Mavridis, 1977; Ahmed, 1983). The double bond N=C $= 1\cdot27$ (1) Å has the usual value in this type of compound and does not merit special comment.

Table 1. *Fractional atomic coordinates* $(\times 10^4)$ *and equivalent isotropic U values* (Å^2 $\times 10^3$) *with e.s.d.'s in parentheses*

Only the H atom of the hydroxyl group (coordinates $\times 10^3$) is included.

$$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	U_{eq}/U
N(1)	8542 (2)	2284 (6)	9901 (9)	41 (2)
N(2)	8634 (2)	4099 (6)	7146 (9)	43 (2)
O	8451 (2)	0313 (6)	12954 (8)	55 (2)
C(1)	9304 (3)	2024 (7)	12615 (9)	41 (2)
C(2)	8985 (3)	0866 (7)	13687 (10)	43 (2)
C(3)	9237 (4)	0272 (9)	15543 (11)	56 (3)
C(4)	9795 (4)	0815 (9)	16341 (12)	57 (3)
C(5)	10107 (3)	1953 (11)	15286 (13)	61 (3)
C(6)	9870 (3)	2564 (10)	13448 (11)	54 (2)
C(7)	9064 (3)	2675 (8)	10674 (10)	41 (2)
C(8)	8324 (3)	2947 (7)	7973 (10)	40 (2)
C(9)	7796 (3)	2315 (7)	7093 (11)	41 (2)
C(10)	7583 (3)	2890 (8)	5170 (11)	44 (2)
C(11)	7905 (3)	4902 (8)	4293 (11)	45 (2)
C(12)	8424 (3)	4665 (7)	5286 (10)	43 (2)
C(13)	7023 (4)	2254 (10)	4090 (15)	63 (3)
C(14)	8785 (4)	5935 (9)	4325 (14)	57 (3)
H	831 (3)	073 (7)	1201 (10)	55

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

C(1)—C(2)	1·41 (1)	N(1)—C(8)	1·43 (1)
C(2)—O	1·34 (1)	C(8)—C(9)	1·38 (1)
C(2)—C(3)	1·39 (1)	C(9)—C(10)	1·39 (1)
C(3)—C(4)	1·39 (1)	C(10)—C(13)	1·50 (1)
C(4)—C(5)	1·38 (1)	C(10)—C(11)	1·38 (1)
C(5)—C(6)	1·37 (1)	C(11)—C(12)	1·38 (1)
C(6)—C(1)	1·41 (1)	C(12)—N(2)	1·35 (1)
C(1)—C(7)	1·45 (1)	C(12)—C(14)	1·49 (1)
C(7)—N(1)	1·27 (1)	N(2)—C(8)	1·32 (1)
O—H	0·76 (6)		

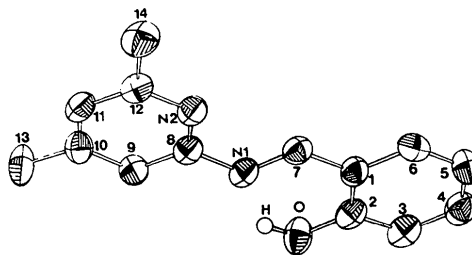| C(2)—C(1)—C(6)	119·4 (6)	N(1)—C(8)—C(9)	116·6 (6)
C(1)—C(2)—C(3)	119·1 (6)	N(1)—C(8)—N(2)	118·8 (5)
C(3)—C(2)—O	119·4 (6)	C(9)—C(8)—N(2)	124·7 (6)
C(1)—C(2)—O	121·6 (5)	C(8)—N(2)—C(12)	117·0 (5)
C(2)—C(3)—C(4)	120·8 (7)	N(2)—C(12)—C(11)	122·0 (6)
C(3)—C(4)—C(5)	120·0 (7)	N(2)—C(12)—C(14)	117·0 (6)
C(4)—C(5)—C(6)	120·7 (7)	C(11)—C(12)—C(14)	121·0 (6)
C(5)—C(6)—C(1)	120·1 (7)	C(12)—C(11)—C(10)	120·6 (6)
C(7)—C(1)—C(2)	121·2 (6)	C(11)—C(10)—C(9)	117·5 (6)
C(7)—C(1)—C(6)	119·4 (6)	C(11)—C(10)—C(13)	120·8 (6)
C(1)—C(7)—N(1)	122·2 (6)	C(9)—C(10)—C(13)	121·7 (6)
C(7)—N(1)—C(8)	120·4 (5)	C(10)—C(9)—C(8)	118·2 (6)
C(2)—O—H	115·6 (43)		

Fig. 1. Molecule showing atomic labelling, and 50% probability thermal ellipsoids. The omitted H atoms have the same numbering as the parent atoms.

Bond lengths and valence angles of the heterocycle are in the range of values found in pyridine rings (Van Havere, Lenstra, Geise, Van den Berg & Benschop, 1982) with N—C values of 1·32 (1) and 1·35 (1) Å. The exocyclic C—N bond of 1·43 (1) Å compares well with the corresponding value of 1·42 (1) Å in the molecule of salampy. The C—CH$_3$ distances in the substituted pyridine have values of 1·49 (1) and 1·50 (1) Å in agreement with the sp^2–sp^3 character of this bond.

The phenyl ring in saladimpy is planar [$\chi^2 = \sum(\Delta/\sigma)^2 = 0·2$] as for salampy, and the exocyclic C(7) lies in this plane [deviation 0·006 (7) Å]. The pyridine ring is also planar ($\chi^2 = 5·0$). The maximum deviation from the plane corresponds to C(9) at −0·008 (6) Å. The two substituents omitted from the calculations of the least-squares plane are out of it by 0·015 (9) and 0·027 (8) Å respectively. The hydrogen-bonded ring C(1),C(2),C(7),O,N(1) deviates markedly from planarity with $\chi^2 = 39·4$ ($\chi^2 = 12·0$ for salampy). The mean planes of the phenyl ring and the hydrogen-bonded ring are coplanar [dihedral angle 1·0 (9)°], while the pyridine ring and the phenyl ring form an angle of 5·4 (9)°. This angle for salampy is 9·4 (9)°.

The tilting and folding of the molecule are revealed by the calculated torsion angles C(9)—C(8)—N(1)—C(7) = 171·5 (6), N(2)—C(8)—N(1)—C(7) = −8·1 (9) and C(1)—C(7)—N(1)—C(8) = −179·6 (6)°. The first two values indicate a rotation of the pyridine ring about the N(1)—C(8) bond by approximately 8° with respect to the rest of the molecule. The last value measures the folding of the molecule about the N(1)—C(7) bond, which is not significant in this case. The molecule of salampy is tilted by approximately 12° about N(1)—C(8) and folded by 178·0 (8)°. The crystal packing is effected only by van der Waals forces.

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Low-Temperature (163 K) Structure of 1,8-Naphthyridine, C₈H₆N₂

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Abstract. $M_r = 130·15$, monoclinic, $P2_1/c$, $a = 6·135$ (6), $b = 10·407$ (8), $c = 11·258$ (9) Å, $\beta = 117·76$ (4)°, $V = 636·1$ (10) Å^3, $Z = 4$, $D_x = 1·359$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·7107$ Å, $\mu = 0·79$ cm^{-1}, $F(000) = 272$, $T = 163$ K. The crystal structure of 1,8-naphthyridine has been re-examined at low temperature (163 K) with the hope of obtaining more reliable information about the observed distortions of the molecule from planarity. A deformation density map has been calculated by opportunely selecting the Fourier coefficients. Full-matrix least-squares refinement converged at R and R_w of 0·049 and 0·053, respectively, for 1275 observed reflections [$I \geq 3\sigma(I)$]. The non-equivalence of the two condensed rings in