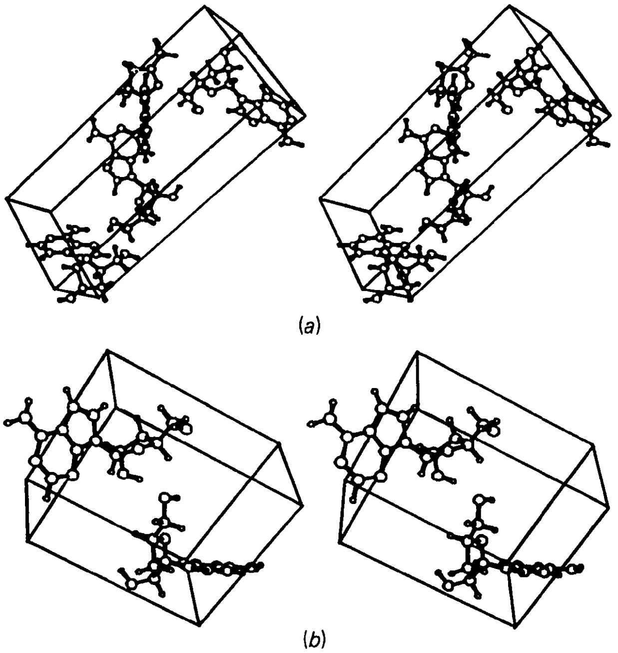

Fig. 2. Unit-cell packing diagrams for (*a*) 3-deaza-3-deoxyadenosine, (*b*) 3′-deoxytubercidin.

Sundaralingam, 1972), which is somewhat higher than for the other related nucleosides. For the majority of modified nucleosides listed in Table 3, including 3′-dTu, the conformation about the C4′—C5′ bond is *gauche*(+)–*trans*. 3′-ddA is exceptional here in having a *gauche*(+)–*gauche*(−) conformation about this bond, which is normally the more stable conformation for nucleosides.

Hydrogen bonding

Fig. 2 shows stereoviews of the packing diagrams of the molecules in their crystal lattice. Hydrogen-bond lengths and angles have been deposited. There is a lack of base–base hydrogen bonds in both crystal lattices as compared with most nucleosides in the solid state.

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Structure of 2-Methoxy-*N*-(4-nitrobenzylidene)-5-pyridylamine

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Abstract. $C_{13}H_{11}N_3O_3$, $M_r = 257{\cdot}2$, monoclinic, $P2_1/n$, $a = 11{\cdot}080\,(2)$, $b = 3{\cdot}376\,(1)$, $c = 14{\cdot}364\,(2)$ Å, $\beta = 112{\cdot}56\,(1)°$, $V = 1231{\cdot}1\,(6)$ Å^3, $Z = 4$, $D_x = 1{\cdot}388$, $D_m = 1{\cdot}385$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}62$ cm^{-1}, $F(000) = 536$, $T = 293$ K, $R = 0{\cdot}0374$ for 1554 observed reflections. The molecule as a whole is in a nearly planar conformation. The dihedral angle between the plane through C(1), C(7), N(1) and C(8) and the phenyl ring is 3·2 (2)°, and that between the phenyl and pyridine rings is 19·6 (2)°. Bond distances indicate a quinoid resonance contribution throughout the molecule.

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Introduction. As part of our ongoing studies of the effects of aromatic substitution on the optical or electro-optical properties of Schiff bases we present this report on the centrosymmetric structure of the Schiff base of 4-nitrobenzaldehyde with 2-methoxy-5-pyridylamine.

Experimental. The title compound was prepared by condensation of the corresponding aldehyde and amine in ethanol. Diamond-shaped crystals were grown by evaporation of ethanol solutions. Density measured by flotation in aqueous $ZnCl_2$. A summary of data collection and structure refinement is given in Table 1. Intensities were corrected for Lorentz and polarization effects.

Scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974). Structure solved and refined with *SHELX76* (Sheldrick, 1976). Refinement by full-matrix weighted least squares [$\sum w(F_o - F_c)^2$ minimized, $w = 1/(\sigma_F^2 + 0{\cdot}001F^2)$]. Best *E* map ($E > 1{\cdot}2$) revealed all non-H atoms. H atoms were located at the end of isotropic refinement in an electron-density difference map. Final refinement of the positional parameters of all atoms and anisotropic thermal parameters for non-H atoms with fixed isotropic thermal parameters for the H atoms (1·25 times those of the atoms to which they were bonded) led to $R = 0{\cdot}0374$.

Discussion. Geometry and atom labelling of the molecule are shown in Fig. 1. Final coordinates and equivalent isotropic temperature factors are listed in

Table 1. *Summary of data collection and structure refinement*

(*a*) Data collection (293 K)*	
Radiation	Mo *K*α (Zr-filtered)
Mode	θ–2θ scan
Scan range	1·8° plus α_1–α_2 divergence in 2θ
Scan rate (° min⁻¹)	1·0–10·0
Background	¼ of scan time at start and end of each scan
2θ range (°)	2·5–47·0
Range in *h, k, l*: min.	0, 0, 16
max.	12, 9, 14
Total reflections measured	2094
Number of unique reflections	1833
Crystal dimensions (mm)	0·1 × 0·4 × 0·5
Absorption correction	None
(*b*) Structure refinement	
Reflections used ($F > 5\sigma_F$)	1554
Number of variables	205
Goodness of fit	1·5
R, wR	0·0374, 0·0542
R for all data	0·0438
Max. shift/e.s.d.	0·041
Max. density in difference map (e Å^{-3})	0·15
Min. density in difference map (e Å^{-3})	−0·20

* Unit-cell parameters were obtained by least-squares refinement of 14 reflections with $20 < 2\theta < 22°$. Syntex $P2_1$ autodiffractometer. Crystal stability and X-ray damage were monitored by measurement of three check reflections every 67 reflections. No damage to the crystal was observed.

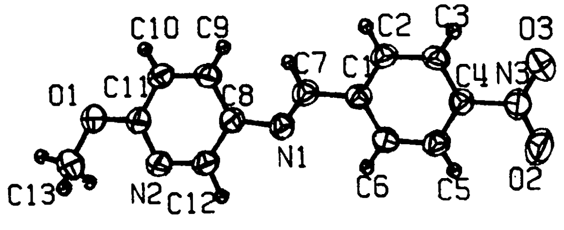

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule showing the atomic labelling.

Table 2. *Coordinates for non-*H *atoms* (×10⁴) *and equivalent isotropic temperature factors* (Å² ×10⁴)

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	*y*	*z*	U_{eq}
C(1)	4361 (1)	3166 (2)	337 (1)	433 (8)
C(2)	3269 (1)	2810 (2)	−524 (1)	501 (9)
C(3)	3390 (1)	1985 (2)	−1314 (1)	469 (9)
C(4)	4620 (1)	1529 (2)	−1229 (1)	443 (8)
C(5)	5732 (2)	1890 (2)	−396 (1)	489 (9)
C(6)	5594 (1)	2708 (2)	387 (1)	459 (8)
C(7)	4196 (1)	4040 (2)	1163 (1)	493 (9)
C(8)	4914 (1)	5194 (2)	2755 (1)	454 (8)
C(9)	3735 (2)	5275 (2)	2892 (1)	507 (9)
C(10)	3675 (2)	6107 (2)	3685 (1)	578 (10)
C(11)	4798 (1)	6851 (2)	4339 (1)	486 (9)
C(12)	5968 (1)	5962 (2)	3456 (1)	520 (9)
C(13)	5861 (2)	8412 (3)	5815 (2)	733 (13)
N(1)	5135 (1)	4354 (2)	1981 (1)	483 (8)
N(2)	5932 (1)	6798 (2)	4242 (1)	519 (8)
N(3)	4755 (1)	623 (2)	−2059 (1)	585 (9)
O(1)	4699 (1)	7652 (2)	5126 (1)	677 (8)
O(2)	5843 (1)	461 (2)	−2060 (1)	941 (11)
O(3)	3776 (1)	101 (2)	−2718 (1)	900 (10)

Table 3. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·391 (2)	C(12)–N(2)	1·342 (1)
C(1)–C(6)	1·394 (2)	C(13)–O(1)	1·436 (1)
C(1)–C(7)	1·464 (2)	N(3)–O(2)	1·214 (2)
C(2)–C(3)	1·377 (2)	N(3)–O(3)	1·215 (1)
C(3)–C(4)	1·375 (2)	C(2)–H(C2)	0·94 (2)
C(4)–C(5)	1·382 (2)	C(3)–H(C3)	0·91 (2)
C(4)–N(3)	1·468 (2)	C(5)–H(C5)	0·88 (2)
C(5)–C(6)	1·374 (2)	C(6)–H(C6)	0·98 (2)
C(7)–N(1)	1·263 (2)	C(7)–H(C7)	0·99 (2)
C(8)–N(1)	1·415 (2)	C(9)–H(C9)	0·91 (2)
C(8)–C(9)	1·396 (2)	C(10)–H(C10)	0·90 (2)
C(8)–C(12)	1·374 (2)	C(12)–H(C12)	1·00 (2)
C(9)–C(10)	1·359 (2)	C(13)–H1(Me)	1·02 (2)
C(10)–C(11)	1·386 (2)	C(13)–H2(Me)	1·02 (2)
C(11)–O(1)	1·354 (1)	C(13)–H3(Me)	0·90 (2)
C(11)–N(2)	1·317 (1)		
C(2)–C(1)–C(6)	119·2 (1)	N(1)–C(8)–C(12)	117·0 (1)
C(2)–C(1)–C(7)	119·4 (1)	C(9)–C(8)–C(12)	116·9 (1)
C(6)–C(1)–C(7)	121·4 (1)	C(8)–C(9)–C(10)	119·5 (1)
C(1)–C(2)–C(3)	120·9 (1)	C(9)–C(10)–C(11)	118·6 (1)
C(2)–C(3)–C(4)	118·2 (1)	C(10)–C(11)–O(1)	116·7 (1)
C(3)–C(4)–C(5)	122·6 (1)	C(10)–C(11)–N(2)	123·9 (1)
C(3)–C(4)–N(3)	118·6 (1)	O(1)–C(11)–N(2)	119·4 (1)
C(5)–C(4)–N(3)	118·8 (1)	C(11)–N(2)–C(12)	116·3 (1)
C(4)–C(5)–C(6)	118·4 (1)	C(8)–C(12)–N(2)	124·7 (1)
C(1)–C(6)–C(5)	120·6 (1)	C(11)–O(1)–C(13)	117·2 (1)
C(1)–C(7)–N(1)	122·9 (1)	O(2)–N(3)–C(4)	118·0 (1)
C(7)–N(1)–C(8)	120·3 (1)	O(3)–N(3)–C(4)	118·7 (1)
N(1)–C(8)–C(9)	126·1 (1)	O(2)–N(3)–O(3)	123·3 (1)

Table 2.* Table 3 contains the bond lengths and angles with their e.s.d.'s. The bond distances and angles of the title compound are in very good agreement with those of *N*-(4-nitrobenzylidene)-4-dimethylaminoaniline (Nakai, Shiro, Ezumi, Sakata & Kubota, 1976). In both compounds the bond distances C(1)—C(7) and N(1)—C(8) are shortened and N(1)—C(7) lengthened in comparison with those of *N*-benzylideneaniline (1·496, 1·460 and 1·237 Å respectively) (Bürgi & Dunitz, 1970). This trend is attributed to the quinoid resonance forms that result in intramolecular charge transfer among the electron-donating methoxy or dimethylamino groups and the strong electron-withdrawing nitro group. The same effect is responsible for the shortening of the bond length of the nitro group to the phenyl ring. In the absence of electron-donating substituents this bond is about 1·48 Å (Mak & Trotter, 1965).

The quinoid resonance forms noted above result in near planarity of the molecule. The dihedral angle between the phenyl ring and the plane through C(1), C(7), N(1) and C(8) is 3·2 (2)°, whilst that between the phenyl and pyridine rings is 19·6 (2)°. This twist of the pyridine ring around the N(1)—C(8) bond and the deformation of N(1)—C(8)—C(9) to 126·1 (1)° minimizes the interaction between H(C7) and H(C9) (Moustakali-Mavridis, Hadjoudis & Mavridis, 1980; Nakai *et al.*, 1976).

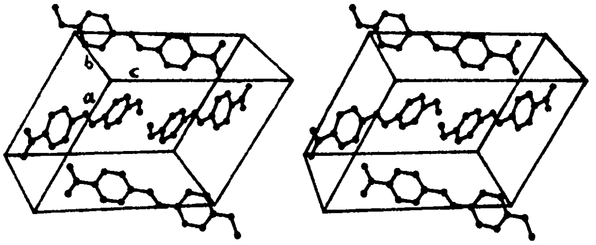

Fig. 2. Stereoscopic view of the molecular packing as viewed down the *b* axis (*ORTEP*; Johnson, 1965).

The packing diagram is shown in Fig. 2. Intermolecular contacts are normal van der Waals interactions. The distance between adjacent molecular planes related by inversion centres is 3·53 (2) Å.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43980 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Tris(diphenylphosphino)amine

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Abstract. $C_{36}H_{30}NP_3$, $M_r = 569{\cdot}57$, monoclinic, $I2/a$ (standard space group $C2/c$), $a = 20{\cdot}005\ (4)$, $b = 11{\cdot}350\ (2)$, $c = 26{\cdot}313\ (11)$ Å, $\beta = 92{\cdot}86\ (2)°$, $V = 5967\ (7)$ Å^3, $Z = 8$, $D_x = 1{\cdot}27$ g cm^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}70926$ Å, $\mu = 2{\cdot}19$ cm^{-1}, $F(000) = 2384$, room temperature; final $R = 0{\cdot}057$ for 4432 reflections (including 20 unobserveds). The central N atom is surrounded by three P atoms in an almost planar trigonal arrangement. The mean N—P distance is 1·740 Å and the mean P—N—P angle is 119·2°. The phenyl rings

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