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6-Methoxy-2-[tri(2-pyridyl)methyliminomethyl]phenol

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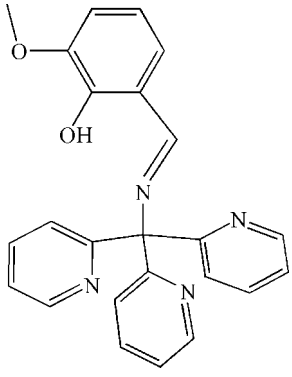
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.113; data-to-parameter ratio = 13.8.

The title compound, $C_{24}H_{20}N_4O_2$, is an unsymmetrical Schiff base which has potential as a hexadentate ligand. There is a relatively strong O—H···N intramolecular hydrogen bond. The X-ray crystallographic results show that, despite the presence of the N_4O_2 group, the title compound does not exhibit a conformation conducive to hexadentate binding. The three pyridine rings show a propeller-like arrangement.

Related literature

For related literature, see: Arnold *et al.* (1998, 2003); Chen *et al.* (2006); Karacan & Somer (2004); Koizumi *et al.* (2005); Ni & Wang (2007); Zhang *et al.* (2003).



Experimental

Crystal data

$C_{24}H_{20}N_4O_2$
M_r = 396.44
Triclinic, $P\overline{1}$
a = 8.8601 (17) Å
b = 10.706 (2) Å
c = 11.147 (2) Å
α = 80.240 (8)°
β = 78.177 (8)°
γ = 87.504 (9)°
V = 1019.9 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 273 (2) K
0.20 × 0.16 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.982, T_{max} = 0.994
8063 measured reflections
3737 independent reflections
3299 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.113
S = 1.01
3737 reflections
271 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.86	1.801	2.581 (2)	150 (1)

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Sheldrick, 1998); software used to prepare material for publication: *SHELXL97* and *XP*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2160).

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supplementary materials

Acta Cryst. (2007). E**63**, o4052 [**doi:10.1107/S1600536807044224**]

6-Methoxy-2-[tri(2-pyridyl)methyliminomethyl]phenol

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Comment

Recently, Schiff base ligands have been widely used to assemble alkoxo- or phenoxo-bridged clusters and polymers with novel topological structures and interesting magnetic, catalytic and photochemical properties. (Koizumi *et al.*, 2005; Chen *et al.*, 2006; Karacan & Somer, 2004).

To date, many symmetrical and unsymmetrical Schiff bases with various coordination dentates have been synthesized (Arnold *et al.*, 2003). Herein, we report a new unsymmetrical Schiff basel (I), which possesses an O_2N_4 donor set affording a potentially hexadentate ligand.

The geometry and labeling scheme for (I) are shown in Figure 1. The imide bond length of 1.2706 (17) Å for N1—C9 is slightly shorter than that of found in 6-Methoxy-2-[2-pyridylmethyliminomethyl]phenol (1.278 (3) Å) (Ni & Wang, 2007) and very similar to that of 4-Bromo-2-(2-pyridylmethyliminomethyl)phenol (1.269 (4) Å) (Zhang *et al.*,2003). There is a relatively strong intramolecular N···H—O bond in (I) with N···O1 at 2.581 (2) Å and N1···H1—O1 at 150.41 (12)° which is similar to what was found in related structures (Ni & Wang, 2007; Zhang *et al.*, 2003).

It is noteworthy that the conformation of the three pyridine rings in (I) are significantly different from those in metal-organic complexes $[Cu(NO_2)_2(tpmbz)]$ and $[\{Cu(NO_2)(tpmsal)\}_2]\cdot Et_2O$ in which three pyridine rings chelate the Cu(II) ions as tripodal ligands (Arnold *et al.*, 2003) indicating that (I) is not a good hexadentate candidate in its present conformation.

Experimental

The material 1,1,1-tris(2-pyridyl)methylamine (tpm) was prepared according to the literature (Arnold *et al.*, 1998). The title compound was prepared as follows: tpm (0.02 mol) was added to a stirred ethanol solution of O-vanillin (0.02 mol, 30 ml). The reaction mixture was stirred about 1 h and then the mixture was allowed to stand at room temperature for about two days after which yellow block single cystals were collected. Yield: 60%. Elemental analysis [found (calculated)] for $C_{24}H_{20}N_4O_2$: C 72.82 (72.71), H 4.82 (5.06), N 14.10% (14.13%).

Refinement

H atoms bound to C and O atoms were visible in difference maps and were placed using the HFIX commands in *SHELXL-97* and refined with a riding model (C—H 0.97 Å or C—H 0.93 Å, and O—H 0.85 Å) with the constraint U_{iso}(H) = 1.5U_{eq}(methyl carrier), 1.5U_{eq}(O) and U_{iso}(H) = 1.2Ueq(C) for all other H atoms.

Figures

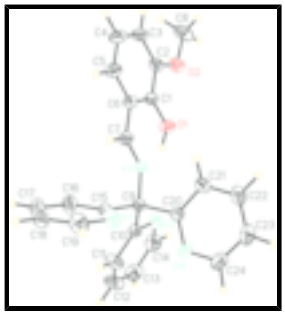


Fig. 1. A view of (I) with the unique atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

6-Methoxy-2-[tri(2-pyridyl)methyliminomethyl]phenol

Crystal data

$C_{24}H_{20}N_4O_2$	$Z = 2$
$M_r = 396.44$	$F_{000} = 416$
Triclinic, $P\overline{1}$	$D_x = 1.291$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.8601$ (17) Å	Cell parameters from 3737 reflections
$b = 10.706$ (2) Å	$\theta = 1.9$–25.5º
$c = 11.147$ (2) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 80.240$ (8)º	$T = 273$ (2) K
$\beta = 78.177$ (8)º	Block, yellow
$\gamma = 87.504$ (9)º	0.20 × 0.16 × 0.10 mm
$V = 1019.9$ (3) Å^3	

Data collection

Bruker APEXII CCD area-detector diffractometer	3737 independent reflections
Radiation source: fine-focus sealed tube	3299 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.018$
$T = 293$(2) K	$\theta_{max} = 25.5$º
φ and ω scans	$\theta_{min} = 1.9$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -10 \rightarrow 9$
$T_{min} = 0.982$, $T_{max} = 0.994$	$k = -12 \rightarrow 9$
8063 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters not refined
$wR(F^2) = 0.113$	$w = 1/[\sigma^2(F_o^2) + (0.0738P)^2 + 0.2154P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
3737 reflections	$\Delta\rho_{max} = 0.20$ e Å^{-3}
271 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.33266 (12)	0.74242 (10)	1.02986 (9)	0.0385 (2)
C6	0.32798 (15)	0.75382 (12)	1.24160 (11)	0.0411 (3)
C7	0.40038 (15)	0.77263 (13)	1.11077 (12)	0.0420 (3)
H7A	0.4984	0.8075	1.0856	0.050*
C16	0.66470 (16)	0.68999 (16)	0.91031 (14)	0.0561 (4)
H16A	0.6218	0.6133	0.9528	0.067*
C13	0.2638 (2)	0.46101 (15)	0.74375 (17)	0.0654 (4)
H13A	0.2294	0.3947	0.7124	0.078*
C17	0.82107 (19)	0.7082 (2)	0.88838 (17)	0.0725 (5)
H17A	0.8847	0.6436	0.9172	0.087*
O1	0.09906 (11)	0.66779 (10)	1.20396 (8)	0.0502 (3)
O2	−0.03696 (12)	0.64400 (11)	1.43905 (9)	0.0589 (3)
C1	0.17907 (15)	0.70342 (12)	1.28201 (11)	0.0395 (3)
C9	0.39813 (13)	0.76771 (11)	0.89521 (10)	0.0351 (3)
C15	0.57358 (14)	0.78706 (12)	0.86825 (11)	0.0386 (3)
C20	0.31630 (13)	0.88584 (11)	0.83720 (11)	0.0362 (3)
C10	0.35609 (14)	0.65406 (12)	0.84055 (11)	0.0381 (3)
N4	0.32578 (15)	0.89811 (11)	0.71510 (10)	0.0499 (3)
C2	0.10966 (16)	0.69167 (13)	1.40953 (12)	0.0464 (3)
N3	0.63130 (14)	0.89841 (12)	0.80865 (13)	0.0563 (3)
C3	0.1897 (2)	0.72685 (15)	1.49179 (13)	0.0580 (4)
H3B	0.1440	0.7183	1.5756	0.070*
N2	0.21454 (13)	0.60759 (12)	0.88633 (12)	0.0518 (3)
C5	0.40627 (18)	0.78922 (15)	1.32790 (13)	0.0546 (4)

H5A	0.5050	0.8224	1.3012	0.066*
C21	0.23894 (17)	0.97280 (14)	0.90640 (14)	0.0511 (3)
H21A	0.2338	0.9618	0.9917	0.061*
C4	0.3381 (2)	0.77516 (16)	1.45114 (14)	0.0620 (4)
H4B	0.3912	0.7979	1.5079	0.074*
C23	0.17926 (19)	1.09068 (15)	0.72196 (17)	0.0609 (4)
H23A	0.1336	1.1598	0.6799	0.073*
C24	0.2584 (2)	1.00038 (15)	0.65985 (15)	0.0610 (4)
H24A	0.2658	1.0105	0.5743	0.073*
C18	0.88412 (17)	0.8203 (2)	0.82472 (17)	0.0666 (5)
H18A	0.9901	0.8327	0.8080	0.080*
C14	0.1720 (2)	0.51215 (16)	0.83700 (17)	0.0611 (4)
H14A	0.0739	0.4789	0.8684	0.073*
C19	0.78688 (18)	0.91319 (17)	0.78668 (18)	0.0666 (5)
H19A	0.8283	0.9902	0.7437	0.080*
C22	0.16936 (18)	1.07631 (15)	0.84721 (17)	0.0615 (4)
H22A	0.1162	1.1357	0.8923	0.074*
C11	0.4556 (2)	0.60833 (15)	0.74578 (14)	0.0599 (4)
H11A	0.5530	0.6430	0.7148	0.072*
C12	0.4077 (2)	0.50963 (17)	0.69741 (17)	0.0738 (5)
H12A	0.4732	0.4766	0.6337	0.089*
C8	−0.1187 (2)	0.6382 (2)	1.56427 (16)	0.0797 (6)
H8A	−0.2192	0.6035	1.5727	0.120*
H8B	−0.1290	0.7219	1.5852	0.120*
H8C	−0.0630	0.5851	1.6191	0.120*
H1	0.1579	0.6804	1.1318	0.120*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0365 (5)	0.0469 (6)	0.0328 (5)	−0.0028 (4)	−0.0057 (4)	−0.0091 (4)
C6	0.0468 (7)	0.0432 (7)	0.0350 (6)	−0.0018 (5)	−0.0100 (5)	−0.0084 (5)
C7	0.0380 (6)	0.0491 (7)	0.0397 (7)	−0.0070 (5)	−0.0067 (5)	−0.0094 (5)
C16	0.0411 (7)	0.0637 (9)	0.0540 (8)	0.0056 (6)	−0.0037 (6)	0.0082 (7)
C13	0.0937 (13)	0.0435 (8)	0.0690 (10)	−0.0053 (8)	−0.0311 (9)	−0.0178 (7)
C17	0.0472 (9)	0.0974 (14)	0.0686 (11)	0.0196 (9)	−0.0127 (8)	−0.0050 (10)
O1	0.0444 (5)	0.0670 (6)	0.0399 (5)	−0.0136 (4)	−0.0046 (4)	−0.0122 (4)
O2	0.0577 (6)	0.0671 (7)	0.0428 (5)	−0.0029 (5)	0.0074 (5)	−0.0042 (5)
C1	0.0463 (7)	0.0374 (6)	0.0347 (6)	0.0014 (5)	−0.0072 (5)	−0.0069 (5)
C9	0.0334 (6)	0.0403 (6)	0.0319 (6)	−0.0014 (5)	−0.0049 (5)	−0.0081 (5)
C15	0.0350 (6)	0.0478 (7)	0.0341 (6)	−0.0002 (5)	−0.0051 (5)	−0.0118 (5)
C20	0.0317 (6)	0.0393 (6)	0.0402 (6)	−0.0033 (5)	−0.0092 (5)	−0.0103 (5)
C10	0.0424 (7)	0.0363 (6)	0.0364 (6)	0.0011 (5)	−0.0099 (5)	−0.0063 (5)
N4	0.0634 (7)	0.0481 (7)	0.0426 (6)	0.0057 (5)	−0.0198 (5)	−0.0101 (5)
C2	0.0548 (8)	0.0417 (7)	0.0376 (7)	0.0041 (6)	−0.0016 (6)	−0.0029 (5)
N3	0.0457 (7)	0.0509 (7)	0.0732 (8)	−0.0057 (5)	−0.0081 (6)	−0.0157 (6)
C3	0.0813 (11)	0.0601 (9)	0.0307 (6)	0.0065 (8)	−0.0076 (7)	−0.0090 (6)
N2	0.0439 (6)	0.0542 (7)	0.0615 (7)	−0.0059 (5)	−0.0114 (5)	−0.0190 (6)

C5	0.0587 (9)	0.0645 (9)	0.0462 (8)	−0.0082 (7)	−0.0171 (7)	−0.0147 (7)
C21	0.0518 (8)	0.0523 (8)	0.0509 (8)	0.0074 (6)	−0.0089 (6)	−0.0167 (6)
C4	0.0817 (11)	0.0700 (10)	0.0422 (8)	−0.0031 (8)	−0.0231 (8)	−0.0178 (7)
C23	0.0581 (9)	0.0444 (8)	0.0852 (12)	0.0050 (7)	−0.0321 (8)	−0.0044 (7)
C24	0.0783 (11)	0.0555 (9)	0.0547 (9)	0.0027 (8)	−0.0313 (8)	−0.0033 (7)
C18	0.0343 (7)	0.0971 (14)	0.0726 (11)	−0.0035 (8)	−0.0057 (7)	−0.0309 (10)
C14	0.0593 (9)	0.0547 (9)	0.0768 (11)	−0.0104 (7)	−0.0225 (8)	−0.0182 (8)
C19	0.0480 (9)	0.0624 (10)	0.0895 (12)	−0.0136 (7)	−0.0047 (8)	−0.0210 (9)
C22	0.0567 (9)	0.0490 (9)	0.0816 (11)	0.0130 (7)	−0.0150 (8)	−0.0201 (8)
C11	0.0651 (10)	0.0586 (9)	0.0536 (8)	−0.0081 (7)	0.0071 (7)	−0.0237 (7)
C12	0.1006 (14)	0.0615 (10)	0.0604 (10)	0.0029 (10)	−0.0011 (9)	−0.0324 (8)
C8	0.0846 (13)	0.0830 (13)	0.0548 (10)	−0.0039 (10)	0.0249 (9)	−0.0112 (9)

Geometric parameters (Å, °)

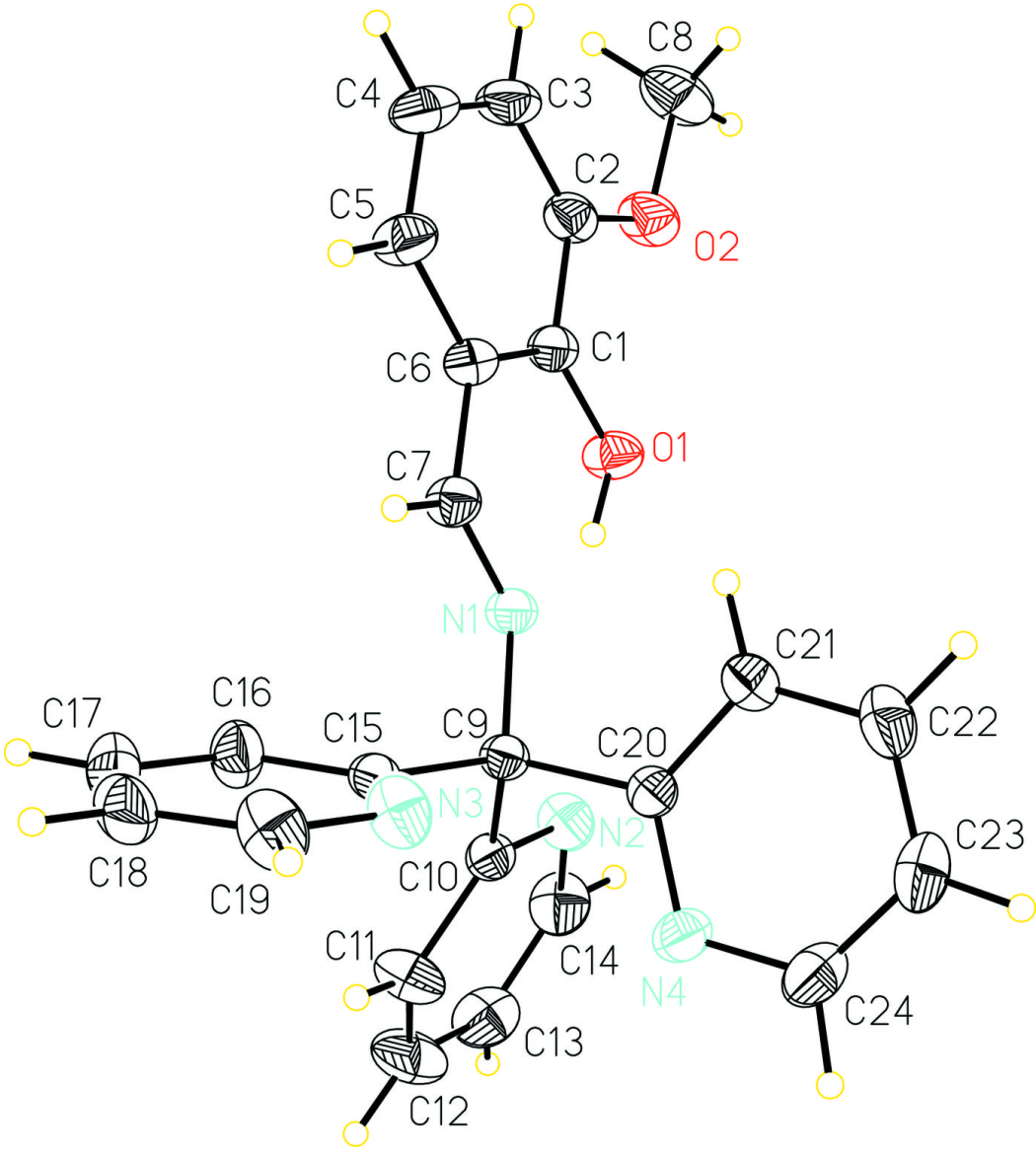
N1—C7	1.2711 (16)	N4—C24	1.3416 (19)
N1—C9	1.4771 (15)	C2—C3	1.376 (2)
C6—C5	1.4037 (18)	N3—C19	1.361 (2)
C6—C1	1.4038 (19)	C3—C4	1.392 (2)
C6—C7	1.4516 (18)	C3—H3B	0.9300
C7—H7A	0.9300	N2—C14	1.3408 (19)
C16—C15	1.3669 (19)	C5—C4	1.368 (2)
C16—C17	1.373 (2)	C5—H5A	0.9300
C16—H16A	0.9300	C21—C22	1.379 (2)
C13—C14	1.360 (2)	C21—H21A	0.9300
C13—C12	1.364 (3)	C4—H4B	0.9300
C13—H13A	0.9300	C23—C22	1.363 (2)
C17—C18	1.365 (3)	C23—C24	1.369 (2)
C17—H17A	0.9300	C23—H23A	0.9300
O1—C1	1.3393 (15)	C24—H24A	0.9300
O1—H1	0.8570	C18—C19	1.357 (3)
O2—C2	1.3718 (18)	C18—H18A	0.9300
O2—C8	1.4272 (18)	C14—H14A	0.9300
C1—C2	1.4152 (18)	C19—H19A	0.9300
C9—C15	1.5379 (16)	C22—H22A	0.9300
C9—C20	1.5421 (17)	C11—C12	1.383 (2)
C9—C10	1.5408 (17)	C11—H11A	0.9300
C15—N3	1.3302 (19)	C12—H12A	0.9300
C20—N4	1.3306 (16)	C8—H8A	0.9600
C20—C21	1.3795 (18)	C8—H8B	0.9600
C10—N2	1.3367 (17)	C8—H8C	0.9600
C10—C11	1.3744 (19)		
C7—N1—C9	122.94 (11)	C2—C3—H3B	119.6
C5—C6—C1	119.81 (12)	C4—C3—H3B	119.6
C5—C6—C7	119.61 (13)	C10—N2—C14	117.38 (13)
C1—C6—C7	120.57 (11)	C4—C5—C6	120.38 (15)
N1—C7—C6	121.38 (12)	C4—C5—H5A	119.8
N1—C7—H7A	119.3	C6—C5—H5A	119.8
C6—C7—H7A	119.3	C20—C21—C22	119.03 (14)

C15—C16—C17	118.52 (15)	C20—C21—H21A	120.5
C15—C16—H16A	120.7	C22—C21—H21A	120.5
C17—C16—H16A	120.7	C5—C4—C3	120.20 (14)
C14—C13—C12	118.17 (14)	C5—C4—H4B	119.9
C14—C13—H13A	120.9	C3—C4—H4B	119.9
C12—C13—H13A	120.9	C22—C23—C24	118.06 (14)
C18—C17—C16	120.67 (16)	C22—C23—H23A	121.0
C18—C17—H17A	119.7	C24—C23—H23A	121.0
C16—C17—H17A	119.7	N4—C24—C23	123.86 (15)
C1—O1—H1	106.3	N4—C24—H24A	118.1
C2—O2—C8	117.61 (13)	C23—C24—H24A	118.1
O1—C1—C6	122.42 (11)	C19—C18—C17	117.75 (15)
O1—C1—C2	118.66 (12)	C19—C18—H18A	121.1
C6—C1—C2	118.92 (12)	C17—C18—H18A	121.1
N1—C9—C15	112.03 (9)	N2—C14—C13	123.90 (16)
N1—C9—C20	108.17 (9)	N2—C14—H14A	118.0
C15—C9—C20	110.75 (10)	C13—C14—H14A	118.0
N1—C9—C10	106.79 (10)	C18—C19—N3	122.84 (16)
C15—C9—C10	111.39 (10)	C18—C19—H19A	118.6
C20—C9—C10	107.49 (9)	N3—C19—H19A	118.6
N3—C15—C16	122.15 (13)	C23—C22—C21	119.38 (14)
N3—C15—C9	119.26 (11)	C23—C22—H22A	120.3
C16—C15—C9	118.55 (12)	C21—C22—H22A	120.3
N4—C20—C21	122.23 (12)	C10—C11—C12	118.59 (15)
N4—C20—C9	115.11 (10)	C10—C11—H11A	120.7
C21—C20—C9	122.66 (11)	C12—C11—H11A	120.7
N2—C10—C11	122.35 (12)	C13—C12—C11	119.60 (15)
N2—C10—C9	115.91 (10)	C13—C12—H12A	120.2
C11—C10—C9	121.65 (12)	C11—C12—H12A	120.2
C20—N4—C24	117.43 (12)	O2—C8—H8A	109.5
O2—C2—C3	125.78 (12)	O2—C8—H8B	109.5
O2—C2—C1	114.39 (12)	H8A—C8—H8B	109.5
C3—C2—C1	119.82 (14)	O2—C8—H8C	109.5
C15—N3—C19	118.03 (14)	H8A—C8—H8C	109.5
C2—C3—C4	120.86 (13)	H8B—C8—H8C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.86	??	2.581 (2)	150 (1)

Fig. 1


C8
C4
C3
C2
C5
O2
C1
C6
O1
C7
N1
C21
C16
C17
C15
C9
C20
C22
N3
N2
C23
C18
C19
C10
C11
C14
N4
C24
C13
C12