

4,6,12,14-Tetra-*tert*-butyl-17-*N*-(6-methylpyridin-2-yl)-8,16-dioxa-17-azatetracyclo-[7.7.1.0^{2,7}.0^{10,15}]heptadeca-2,4,6,10,12,14-hexaene methanol solvate

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Key indicators

Single-crystal X-ray study

$T = 273$ K

Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å

Disorder in main residue

R factor = 0.044

wR factor = 0.082

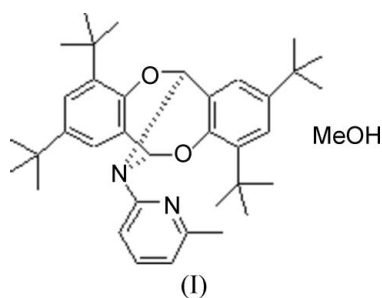
Data-to-parameter ratio = 14.8

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The molecule of the title compound, $\text{C}_{37}\text{H}_{52}\text{N}_2\text{O}_3$, exhibits a propeller-like conformation, influenced by one intramolecular $\text{C}-\text{H}\cdots\text{N}$ and four intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds. In the crystal structure, there are four weak $\text{C}-\text{H}\cdots\pi$ interactions.

Comment

Multidentate Schiff base ligands and their metal complexes have been extensively studied for many years (Daier *et al.*, 2004; Munro & Camp, 2003; Weber, 1967; Xu *et al.*, 2001). Schiff bases can be prepared by condensation of different types of amines and carbonyl compounds; one of the most popular methods has been the use of salicylideneamines, which can serve as efficient models for biologically important systems (Long, 1995) in addition to their diverse chelating ability (Kim *et al.*, 2001). The title compound, (I), was unexpectedly obtained in the reaction of 3,5-di-*tert*-butylsalicylaldehyde with 6-amino-2-methylpyridine.



The title compound, (I), possesses three aryl rings, *viz.* benzene ring 1 (atoms C1–C6; denoted R_1), benzene ring 2 (C10–C15; R_2) and pyridyl ring 3 (N2/C19–C23; R_3). These are connected to the central polyhedron (N1/C9/C18/O1/O2/C3/C4/C10/C11) to give a propeller-like conformation, which is influenced by one intramolecular $\text{C}-\text{H}\cdots\text{N}$ and four intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds (Fig. 3 and Table 1). The dihedral angles for R_1/R_2 , R_1/R_3 and R_2/R_3 are 79.4 (1), 27.8 (1) and 73.0 (1)°, respectively. The bond lengths and angles are unexceptional.

$\text{C}-\text{H}\cdots\pi$ interactions often play an important role in the formation of crystal structures (Low *et al.*, 2004; Sylvestre *et al.*, 2005). In the case of (I), an extended crystal structure (Fig. 4) is formed through weak intermolecular $\text{C}-\text{H}\cdots\pi$ interactions (Table 1).

Experimental

The title compound was synthesized by the reaction of 3,5-di-*tert*-butylsalicylaldehyde and 6-methyl-2-aminopyridine, under a nitrogen

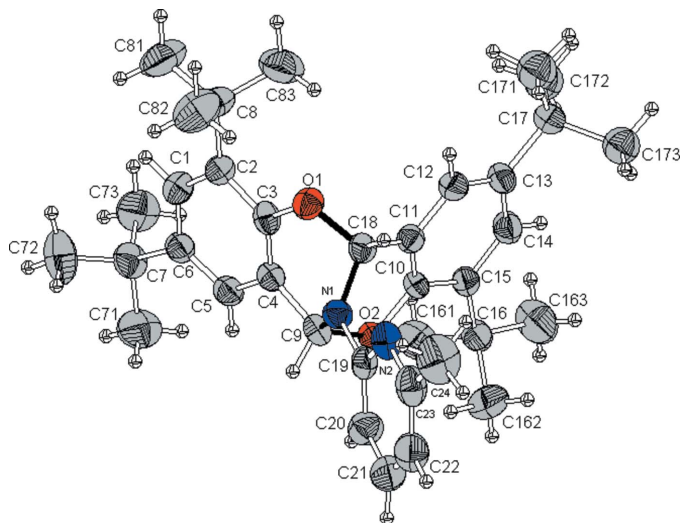


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Only one component of the disordered *tert*-butyl group is shown. The solvent molecule has been omitted for the sake of clarity.

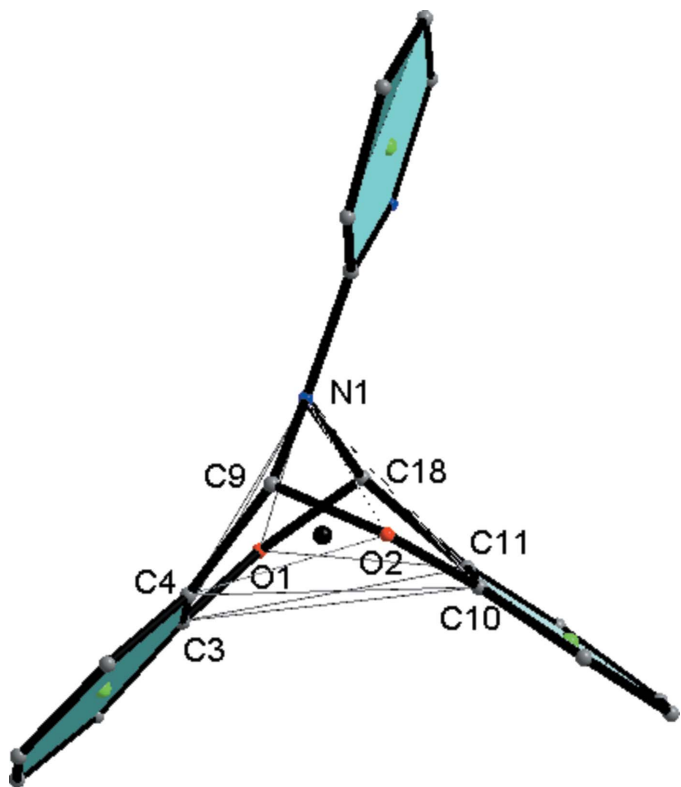


Figure 2
The structure of (I), showing the propeller-like conformation.

atmosphere. A mixture of 3,5-di-*tert*-butylsalicylaldehyde (4 mmol) and 2-amino-6-methylpyridine (4.4 mmol) in ethanol (20 ml) was heated under reflux for 24 h, after adding formic acid as a catalyst. It was then allowed to stand at room temperature overnight. The solid product was isolated by filtration, and purified by thin-layer chromatography. Pale-yellow single crystals suitable for X-ray diffraction were obtained by evaporation of a methanol solution of the product.

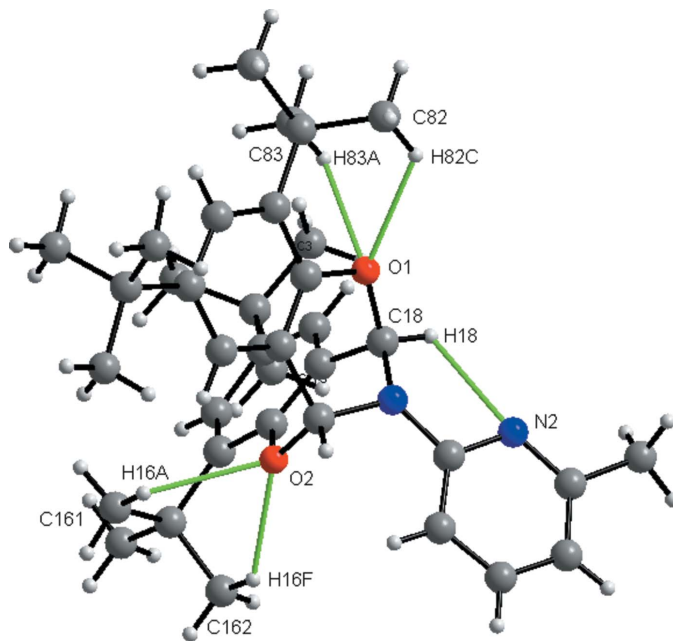


Figure 3
The structure of (I), showing intramolecular hydrogen bonds (indicated by green lines).

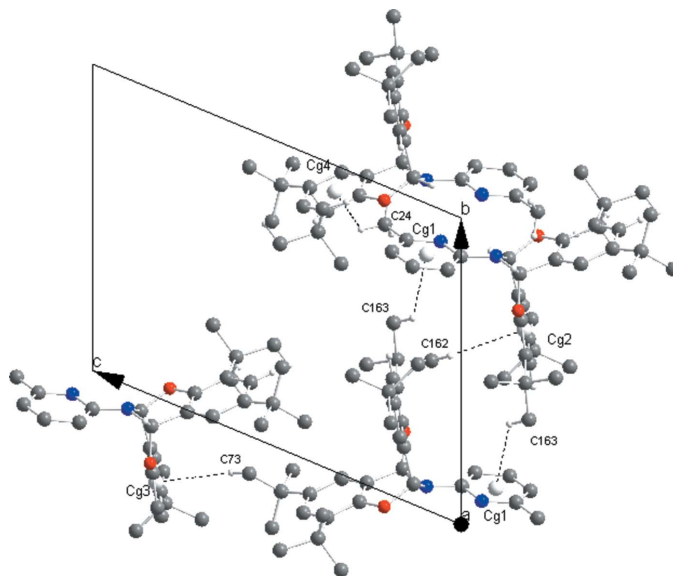


Figure 4
The packing of (I), viewed along the *a* axis, showing the intermolecular C–H··· π interactions as dashed lines.

Crystal data

$C_{36}H_{48}N_2O_2 \cdot CH_4O$
 $M_r = 572.81$
Triclinic, $P\bar{1}$
 $a = 10.282$ (2) Å
 $b = 12.775$ (3) Å
 $c = 15.601$ (3) Å
 $\alpha = 66.313$ (4)°
 $\beta = 82.711$ (4)°
 $\gamma = 68.295$ (4)°
 $V = 1743.0$ (7) Å³

$Z = 2$
 $D_x = 1.091$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 877 reflections
 $\theta = 2.3$ – 18.5 °
 $\mu = 0.07$ mm⁻¹
 $T = 273$ (2) K
Block, pale yellow
0.30 × 0.20 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1913 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.049$
Absorption correction: none	$\theta_{\text{max}} = 25.0^\circ$
9105 measured reflections	$h = -12 \rightarrow 12$
6047 independent reflections	$k = -15 \rightarrow 13$
	$l = -18 \rightarrow 10$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2]$
$wR(F^2) = 0.082$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.62$	$(\Delta/\sigma)_{\text{max}} = 0.015$
6047 reflections	$\Delta\rho_{\text{max}} = 0.27 \text{ e } \text{\AA}^{-3}$
408 parameters	$\Delta\rho_{\text{min}} = -0.32 \text{ e } \text{\AA}^{-3}$

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C18—H18 \cdots N2	0.98	2.36	2.808 (3)	107
C161—H16A \cdots O2	0.96	2.37	3.013 (6)	124
C162—H16F \cdots O2	0.96	2.31	2.967 (8)	125
C82—H82C \cdots O1	0.96	2.40	3.033 (9)	124
C83—H83A \cdots O1	0.96	2.34	2.980 (9)	123
C163—H16I \cdots Cg1 ⁱ	0.96	2.91	3.565 (7)	127
C162—H16D \cdots Cg2 ⁱⁱ	0.96	3.06	3.893 (11)	146
C73—H73A \cdots Cg2 ⁱⁱⁱ	0.96	2.95	3.725 (9)	138
C24—H24B \cdots Cg1 ⁱ	0.96	2.65	3.147 (7)	113

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x-1, y+1, z-1$; (iii) $x-1, y, z$. Cg1 is the centroid of R1 and Cg2 is the centroid of R2

After their location, all H atoms were placed in idealized positions and refined using a riding model; C—H = 0.93–0.98 \AA , O—H = 0.82 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{O})$. One of the *tert*-butyl groups is

disordered over three sites; there are three sets of methyl groups (C171/C172/C173, C174/C175/C176 and C177/C178/C179) with site-occupancy factors of 0.5, 0.25 and 0.25 for groups 1, 2 and 3, respectively.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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