Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 4-[Bis(3,4-dimethoxyphenyl)methyl]-pyridine ethanol monosolvate

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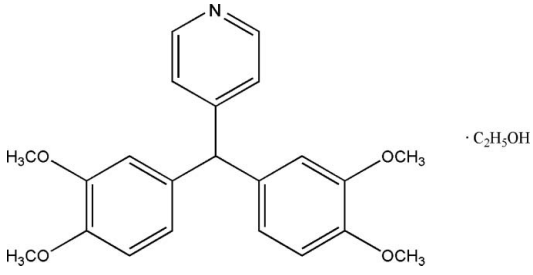
Received 10 May 2010; accepted 12 May 2010

Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.063; $wR$ factor = 0.174; data-to-parameter ratio = 20.1.

In the title compound, $C_{22}H_{23}NO_4\cdot C_2H_6O$, the pyridyl ring is aligned at 89.39 (2) and 87.41 (2)° with respect to the benzene rings, and the three rings connected to the methine C atom are arranged in a propeller-like conformation. The heterocycle is linked to the solvent molecule by an O—H···N hydrogen bond.

## Related literature

For background to the use of pyridine and its derivatives as ligands to bridge different metal ions and form functional coordination compounds, see: Chen *et al.* (2007); Fasina *et al.* (2004); Mancisidor *et al.* (2008). For the synthesis, see: Ostaszewski (1998).




## Experimental

### Crystal data

$C_{22}H_{23}NO_4\cdot C_2H_6O$ $M_r$ = 411.48
Monoclinic, $C2/c$
$a$ = 29.564 (6) Å
$b$ = 8.3810 (17) Å
$c$ = 19.440 (4) Å
$\beta$ = 107.94 (3)°
$V$ = 4582.6 (18) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 295 K
0.27 × 0.20 × 0.19 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
14513 measured reflections
5573 independent reflections
2669 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.042
3 standard reflections every 100 reflections
intensity decay: none

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.174
$S$ = 1.02
5573 reflections
277 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O5—H5···N1 | 0.82 | 2.04 | 2.842 (4) | 167 |

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank the Natural Science Foundation of Shandong Province (No. Z2007B01).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2771).

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# supporting information

*Acta Cryst.* (2010). E**66**, o1378 [https://doi.org/10.1107/S1600536810017460]

## 4-[Bis(3,4-dimethoxyphenyl)methyl]pyridine ethanol monosolvate

**Fang-Fang Jian and Zhi-Peng Ni**

### S1. Comment

Pyridine and its derivatives, are of interest as ligands to bridge different metal ions to form functional coordination compounds, for example: 9,10-bis(4′- pyridylethynyl)-anthracene (Fasina *et al.*, 2004);2,6-bis-(imi-dazol-1 -yl)pyridine (Chen *et al.*, 2007); bis-(pyridine-2-ylmethyl)-benzylamine (Mancisidor *et al.*, 2008). In order to search for new pyridine compounds with higher bioactivity and optical properties, we synthesized the title compound.

In the title compound, the bond lengths and angles are generally normal. The dihedral angles between pyridine ring N1, C20, C19, C18, C22, C21(p1) with C3—C8 (p2) phenyl ring and C10—C15 (p3) phenyl ring are 89.39 (2)° and 87.41 (2)°, the dihedral angles between C3—C8 (p2) phenyl ring and C10—C15 (p3) phenyl ring is 84.33 (2)°, respectively.

The crystal structure is stabilized by intramolecular O—H···N hydrogen bonds (Table 1) and intramolecular C—H···O hydrogen bonds. The donor and acceptor distance are 3.4019Å for C(20) – H(20 A).. O(5) and 3.3902Å for C(21) – H(21 A).. O(3). In addition, there exist four kinds of C—H···Π interaction in the lattice [C2···Cg1=3.441 (2) Å; C3···Cg2=4.052 (3) Å; C17···Cg2=3.774 (2) Å; C24···Cg3=4.187 (1) Å; Cg1, Cg2 and Cg3 refer to pyridine, phenyl C3—C8 and phenyl ring C10—C15, respectively]. In the solid state, all above intermolecular interactions in the title compound stabilize the crystal packing structure.

### S2. Experimental

The title compound was prepared by the reaction of 1,2-dimethoxybenzene (20 mmol), isonicotinaldehyde (40 mmol), and was stirred in dichloromethane solution with 84% sulfuric acid (10 ml) as activator (Ostaszewski *et al.*, 1998). Single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature over a period of 3 days.

### S3. Refinement

H atoms were positioned geometrically and treated as riding on their parent C atoms, with C—H distances in the range 0.93-0.97 Å, and with $U_{iso}$(H)= 1.2-1.5$U_{eq}$ of the parent atoms.

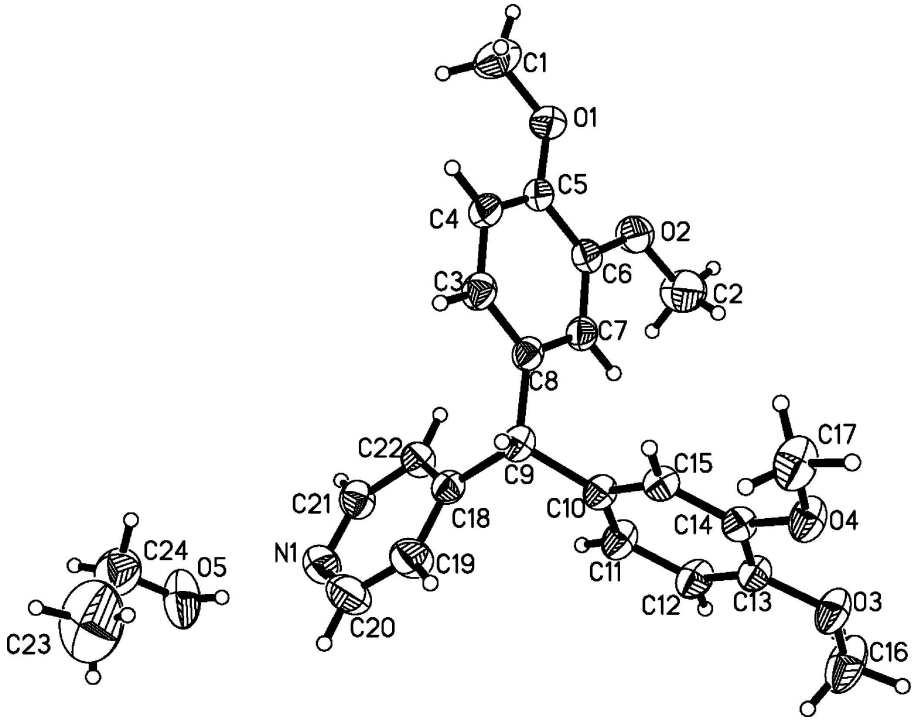


**Figure 1**

The molecular structure of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

**4-[Bis(3,4-dimethoxyphenyl)methyl]pyridine ethanol monosolvate**

*Crystal data*

$C_{22}H_{23}NO_4 \cdot C_2H_6O$
$M_r = 411.48$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 29.564\ (6)$ Å
$b = 8.3810\ (17)$ Å
$c = 19.440\ (4)$ Å
$\beta = 107.94\ (3)°$
$V = 4582.6\ (18)$ Å$^3$
$Z = 8$

$F(000) = 1760$
$D_x = 1.193$ Mg m$^{-3}$
Melting point: 342 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 1.5–25.5°$
$\mu = 0.08$ mm$^{-1}$
$T = 295$ K
Block, colourless
0.27 × 0.20 × 0.19 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
14513 measured reflections
5573 independent reflections
2669 reflections with $I > 2\sigma(I)$

$R_{int} = 0.042$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.5°$
$h = -34 \rightarrow 38$
$k = -11 \rightarrow 10$
$l = -25 \rightarrow 20$
3 standard reflections every 100 reflections
intensity decay: none

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.174$
$S = 1.02$
5573 reflections
277 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.070P)^2 + 0.6422P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å$^{-3}$
$\Delta\rho_{min} = -0.22$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.30498 (6) | 1.1065 (2) | 0.93614 (10) | 0.0848 (6) |
| O2 | 0.23104 (6) | 1.2671 (2) | 0.86968 (10) | 0.0795 (5) |
| O3 | 0.07275 (6) | 1.2438 (2) | 0.48851 (8) | 0.0711 (5) |
| O4 | 0.15262 (5) | 1.1115 (2) | 0.49173 (8) | 0.0648 (5) |
| O5 | 0.02886 (9) | 0.3191 (3) | 0.87938 (15) | 0.1157 (8) |
| H5 | 0.0360 | 0.3742 | 0.8494 | 0.139* |
| N1 | 0.06698 (8) | 0.5258 (3) | 0.79531 (13) | 0.0770 (6) |
| C1 | 0.34474 (13) | 1.0218 (4) | 0.97716 (19) | 0.1275 (14) |
| H1B | 0.3659 | 1.0926 | 1.0109 | 0.191* |
| H1C | 0.3608 | 0.9765 | 0.9458 | 0.191* |
| H1D | 0.3349 | 0.9380 | 1.0030 | 0.191* |
| C2 | 0.19141 (11) | 1.3623 (4) | 0.83401 (19) | 0.1037 (11) |
| H2A | 0.1957 | 1.4676 | 0.8544 | 0.156* |
| H2B | 0.1631 | 1.3158 | 0.8399 | 0.156* |
| H2C | 0.1884 | 1.3683 | 0.7835 | 0.156* |
| C3 | 0.24084 (8) | 0.8041 (3) | 0.80555 (12) | 0.0592 (6) |
| H3A | 0.2443 | 0.6994 | 0.7922 | 0.071* |
| C4 | 0.27668 (8) | 0.8747 (3) | 0.86124 (13) | 0.0627 (7) |
| H4A | 0.3039 | 0.8167 | 0.8845 | 0.075* |
| C5 | 0.27240 (8) | 1.0275 (3) | 0.88221 (12) | 0.0573 (6) |
| C6 | 0.23211 (8) | 1.1147 (3) | 0.84613 (12) | 0.0550 (6) |
| C7 | 0.19656 (8) | 1.0447 (3) | 0.79090 (12) | 0.0546 (6) |
| H7A | 0.1696 | 1.1034 | 0.7672 | 0.066* |
| C8 | 0.20042 (7) | 0.8876 (3) | 0.77007 (11) | 0.0491 (5) |
| C9 | 0.16086 (7) | 0.8074 (3) | 0.71137 (10) | 0.0503 (6) |

| | | | | |
|---|---|---|---|---|
| H9A | 0.1764 | 0.7297 | 0.6884 | 0.060* |
| C10 | 0.13455 (7) | 0.9216 (3) | 0.65203 (11) | 0.0493 (5) |
| C11 | 0.09260 (8) | 0.9934 (3) | 0.64980 (12) | 0.0608 (7) |
| H11A | 0.0783 | 0.9687 | 0.6849 | 0.073* |
| C12 | 0.07107 (8) | 1.1021 (3) | 0.59624 (12) | 0.0626 (7) |
| H12A | 0.0427 | 1.1508 | 0.5960 | 0.075* |
| C13 | 0.09124 (8) | 1.1383 (3) | 0.54365 (11) | 0.0528 (6) |
| C14 | 0.13426 (7) | 1.0673 (3) | 0.54553 (11) | 0.0481 (5) |
| C15 | 0.15522 (7) | 0.9611 (3) | 0.59909 (11) | 0.0486 (5) |
| H15A | 0.1839 | 0.9142 | 0.6002 | 0.058* |
| C16 | 0.02736 (11) | 1.3087 (4) | 0.48086 (18) | 0.1096 (12) |
| H16A | 0.0186 | 1.3801 | 0.4403 | 0.164* |
| H16B | 0.0282 | 1.3659 | 0.5240 | 0.164* |
| H16C | 0.0045 | 1.2240 | 0.4731 | 0.164* |
| C17 | 0.19682 (9) | 1.0437 (3) | 0.49298 (14) | 0.0800 (8) |
| H17A | 0.2065 | 1.0867 | 0.4540 | 0.120* |
| H17B | 0.1935 | 0.9300 | 0.4877 | 0.120* |
| H17C | 0.2203 | 1.0683 | 0.5382 | 0.120* |
| C18 | 0.12750 (8) | 0.7117 (3) | 0.74155 (11) | 0.0517 (6) |
| C19 | 0.09979 (10) | 0.5923 (3) | 0.70163 (13) | 0.0783 (8) |
| H19A | 0.1008 | 0.5715 | 0.6551 | 0.094* |
| C20 | 0.07074 (11) | 0.5034 (4) | 0.72947 (17) | 0.0918 (9) |
| H20A | 0.0527 | 0.4231 | 0.7009 | 0.110* |
| C21 | 0.09305 (9) | 0.6417 (3) | 0.83336 (14) | 0.0657 (7) |
| H21A | 0.0909 | 0.6613 | 0.8793 | 0.079* |
| C22 | 0.12338 (8) | 0.7359 (3) | 0.80920 (12) | 0.0566 (6) |
| H22A | 0.1410 | 0.8157 | 0.8388 | 0.068* |
| C23 | 0.0512 (2) | 0.0797 (6) | 0.8412 (3) | 0.227 (3) |
| H23A | 0.0652 | −0.0205 | 0.8605 | 0.340* |
| H23B | 0.0658 | 0.1179 | 0.8066 | 0.340* |
| H23C | 0.0177 | 0.0656 | 0.8181 | 0.340* |
| C24 | 0.05815 (13) | 0.1877 (5) | 0.8959 (2) | 0.1247 (13) |
| H24A | 0.0909 | 0.2236 | 0.9096 | 0.150* |
| H24B | 0.0534 | 0.1345 | 0.9374 | 0.150* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0727 (12) | 0.0672 (12) | 0.0895 (12) | −0.0157 (10) | −0.0119 (10) | 0.0122 (10) |
| O2 | 0.0763 (13) | 0.0534 (11) | 0.0964 (13) | −0.0007 (9) | 0.0085 (10) | −0.0037 (10) |
| O3 | 0.0654 (11) | 0.0860 (12) | 0.0681 (10) | 0.0276 (10) | 0.0297 (8) | 0.0295 (9) |
| O4 | 0.0664 (11) | 0.0799 (12) | 0.0586 (9) | 0.0179 (9) | 0.0346 (8) | 0.0171 (8) |
| O5 | 0.1304 (19) | 0.0829 (16) | 0.164 (2) | 0.0193 (15) | 0.0893 (17) | 0.0368 (15) |
| N1 | 0.0741 (15) | 0.0770 (16) | 0.0810 (15) | −0.0157 (13) | 0.0257 (12) | 0.0058 (13) |
| C1 | 0.105 (3) | 0.105 (3) | 0.121 (3) | −0.007 (2) | −0.041 (2) | 0.014 (2) |
| C2 | 0.089 (2) | 0.0649 (19) | 0.142 (3) | 0.0179 (17) | 0.013 (2) | −0.011 (2) |
| C3 | 0.0556 (15) | 0.0595 (15) | 0.0647 (14) | 0.0085 (12) | 0.0216 (12) | 0.0095 (12) |
| C4 | 0.0476 (14) | 0.0656 (17) | 0.0708 (16) | 0.0053 (12) | 0.0124 (12) | 0.0195 (14) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C5 | 0.0500 (14) | 0.0582 (16) | 0.0595 (14) | −0.0057 (12) | 0.0107 (11) | 0.0165 (12) |
| C6 | 0.0540 (15) | 0.0517 (15) | 0.0606 (14) | −0.0043 (12) | 0.0193 (12) | 0.0083 (12) |
| C7 | 0.0482 (14) | 0.0567 (15) | 0.0602 (14) | 0.0032 (11) | 0.0185 (11) | 0.0127 (12) |
| C8 | 0.0457 (13) | 0.0568 (15) | 0.0484 (12) | 0.0034 (11) | 0.0198 (10) | 0.0106 (11) |
| C9 | 0.0535 (13) | 0.0549 (14) | 0.0457 (12) | 0.0080 (11) | 0.0201 (10) | 0.0036 (10) |
| C10 | 0.0512 (13) | 0.0551 (14) | 0.0438 (11) | 0.0057 (11) | 0.0179 (10) | 0.0021 (10) |
| C11 | 0.0583 (15) | 0.0809 (18) | 0.0514 (13) | 0.0138 (13) | 0.0289 (11) | 0.0143 (12) |
| C12 | 0.0506 (14) | 0.0802 (18) | 0.0621 (14) | 0.0187 (13) | 0.0247 (12) | 0.0134 (13) |
| C13 | 0.0518 (14) | 0.0593 (14) | 0.0481 (12) | 0.0068 (11) | 0.0163 (10) | 0.0071 (11) |
| C14 | 0.0488 (13) | 0.0541 (13) | 0.0448 (11) | 0.0023 (11) | 0.0194 (10) | −0.0013 (10) |
| C15 | 0.0458 (12) | 0.0539 (14) | 0.0486 (12) | 0.0060 (11) | 0.0185 (10) | −0.0020 (11) |
| C16 | 0.086 (2) | 0.139 (3) | 0.117 (2) | 0.061 (2) | 0.0505 (19) | 0.065 (2) |
| C17 | 0.0815 (19) | 0.100 (2) | 0.0775 (17) | 0.0284 (17) | 0.0530 (15) | 0.0195 (16) |
| C18 | 0.0519 (13) | 0.0559 (14) | 0.0465 (12) | 0.0020 (11) | 0.0141 (10) | 0.0031 (11) |
| C19 | 0.094 (2) | 0.088 (2) | 0.0525 (14) | −0.0251 (17) | 0.0221 (14) | −0.0119 (14) |
| C20 | 0.097 (2) | 0.094 (2) | 0.081 (2) | −0.0386 (18) | 0.0223 (17) | −0.0097 (17) |
| C21 | 0.0691 (17) | 0.0713 (17) | 0.0635 (15) | −0.0018 (15) | 0.0304 (13) | 0.0059 (14) |
| C22 | 0.0624 (15) | 0.0562 (14) | 0.0543 (13) | −0.0049 (12) | 0.0224 (11) | −0.0031 (11) |
| C23 | 0.330 (9) | 0.152 (5) | 0.213 (6) | 0.083 (6) | 0.107 (6) | −0.038 (4) |
| C24 | 0.090 (3) | 0.126 (4) | 0.152 (4) | 0.010 (2) | 0.029 (2) | 0.031 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C5 | 1.358 (3) | C9—H9A | 0.9800 |
| O1—C1 | 1.395 (3) | C10—C11 | 1.367 (3) |
| O2—C6 | 1.361 (3) | C10—C15 | 1.389 (3) |
| O2—C2 | 1.410 (3) | C11—C12 | 1.383 (3) |
| O3—C13 | 1.367 (3) | C11—H11A | 0.9300 |
| O3—C16 | 1.413 (3) | C12—C13 | 1.367 (3) |
| O4—C14 | 1.369 (2) | C12—H12A | 0.9300 |
| O4—C17 | 1.418 (3) | C13—C14 | 1.395 (3) |
| O5—C24 | 1.377 (4) | C14—C15 | 1.365 (3) |
| O5—H5 | 0.8200 | C15—H15A | 0.9300 |
| N1—C21 | 1.317 (3) | C16—H16A | 0.9600 |
| N1—C20 | 1.332 (3) | C16—H16B | 0.9600 |
| C1—H1B | 0.9600 | C16—H16C | 0.9600 |
| C1—H1C | 0.9600 | C17—H17A | 0.9600 |
| C1—H1D | 0.9600 | C17—H17B | 0.9600 |
| C2—H2A | 0.9600 | C17—H17C | 0.9600 |
| C2—H2B | 0.9600 | C18—C19 | 1.373 (3) |
| C2—H2C | 0.9600 | C18—C22 | 1.373 (3) |
| C3—C8 | 1.373 (3) | C19—C20 | 1.368 (4) |
| C3—C4 | 1.392 (3) | C19—H19A | 0.9300 |
| C3—H3A | 0.9300 | C20—H20A | 0.9300 |
| C4—C5 | 1.362 (3) | C21—C22 | 1.381 (3) |
| C4—H4A | 0.9300 | C21—H21A | 0.9300 |
| C5—C6 | 1.389 (3) | C22—H22A | 0.9300 |
| C6—C7 | 1.381 (3) | C23—C24 | 1.362 (5) |

| | | | |
|---|---|---|---|
| C7—C8 | 1.392 (3) | C23—H23A | 0.9600 |
| C7—H7A | 0.9300 | C23—H23B | 0.9600 |
| C8—C9 | 1.517 (3) | C23—H23C | 0.9600 |
| C9—C10 | 1.516 (3) | C24—H24A | 0.9700 |
| C9—C18 | 1.522 (3) | C24—H24B | 0.9700 |
| | | | |
| C5—O1—C1 | 117.8 (2) | C11—C12—H12A | 119.9 |
| C6—O2—C2 | 117.9 (2) | C12—C13—O3 | 124.7 (2) |
| C13—O3—C16 | 117.95 (19) | C12—C13—C14 | 119.4 (2) |
| C14—O4—C17 | 117.27 (18) | O3—C13—C14 | 115.96 (19) |
| C24—O5—H5 | 109.5 | C15—C14—O4 | 124.44 (19) |
| C21—N1—C20 | 115.9 (2) | C15—C14—C13 | 119.63 (19) |
| O1—C1—H1B | 109.5 | O4—C14—C13 | 115.93 (19) |
| O1—C1—H1C | 109.5 | C14—C15—C10 | 121.4 (2) |
| H1B—C1—H1C | 109.5 | C14—C15—H15A | 119.3 |
| O1—C1—H1D | 109.5 | C10—C15—H15A | 119.3 |
| H1B—C1—H1D | 109.5 | O3—C16—H16A | 109.5 |
| H1C—C1—H1D | 109.5 | O3—C16—H16B | 109.5 |
| O2—C2—H2A | 109.5 | H16A—C16—H16B | 109.5 |
| O2—C2—H2B | 109.5 | O3—C16—H16C | 109.5 |
| H2A—C2—H2B | 109.5 | H16A—C16—H16C | 109.5 |
| O2—C2—H2C | 109.5 | H16B—C16—H16C | 109.5 |
| H2A—C2—H2C | 109.5 | O4—C17—H17A | 109.5 |
| H2B—C2—H2C | 109.5 | O4—C17—H17B | 109.5 |
| C8—C3—C4 | 120.7 (2) | H17A—C17—H17B | 109.5 |
| C8—C3—H3A | 119.7 | O4—C17—H17C | 109.5 |
| C4—C3—H3A | 119.7 | H17A—C17—H17C | 109.5 |
| C5—C4—C3 | 120.9 (2) | H17B—C17—H17C | 109.5 |
| C5—C4—H4A | 119.5 | C19—C18—C22 | 115.8 (2) |
| C3—C4—H4A | 119.5 | C19—C18—C9 | 120.7 (2) |
| O1—C5—C4 | 125.5 (2) | C22—C18—C9 | 123.4 (2) |
| O1—C5—C6 | 115.3 (2) | C20—C19—C18 | 120.8 (2) |
| C4—C5—C6 | 119.2 (2) | C20—C19—H19A | 119.6 |
| O2—C6—C7 | 124.7 (2) | C18—C19—H19A | 119.6 |
| O2—C6—C5 | 115.4 (2) | N1—C20—C19 | 123.5 (3) |
| C7—C6—C5 | 119.9 (2) | N1—C20—H20A | 118.2 |
| C6—C7—C8 | 121.1 (2) | C19—C20—H20A | 118.2 |
| C6—C7—H7A | 119.4 | N1—C21—C22 | 124.0 (2) |
| C8—C7—H7A | 119.4 | N1—C21—H21A | 118.0 |
| C3—C8—C7 | 118.2 (2) | C22—C21—H21A | 118.0 |
| C3—C8—C9 | 120.1 (2) | C18—C22—C21 | 120.0 (2) |
| C7—C8—C9 | 121.67 (19) | C18—C22—H22A | 120.0 |
| C10—C9—C8 | 112.84 (19) | C21—C22—H22A | 120.0 |
| C10—C9—C18 | 112.59 (18) | C24—C23—H23A | 109.5 |
| C8—C9—C18 | 112.63 (16) | C24—C23—H23B | 109.5 |
| C10—C9—H9A | 106.0 | H23A—C23—H23B | 109.5 |
| C8—C9—H9A | 106.0 | C24—C23—H23C | 109.5 |
| C18—C9—H9A | 106.0 | H23A—C23—H23C | 109.5 |

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| C11—C10—C15 | 118.3 (2) | H23B—C23—H23C | 109.5 |
| C11—C10—C9 | 123.36 (19) | C23—C24—O5 | 114.7 (4) |
| C15—C10—C9 | 118.31 (19) | C23—C24—H24A | 108.6 |
| C10—C11—C12 | 121.1 (2) | O5—C24—H24A | 108.6 |
| C10—C11—H11A | 119.4 | C23—C24—H24B | 108.6 |
| C12—C11—H11A | 119.4 | O5—C24—H24B | 108.6 |
| C13—C12—C11 | 120.2 (2) | H24A—C24—H24B | 107.6 |
| C13—C12—H12A | 119.9 | | |

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| C8—C3—C4—C5 | 0.5 (3) | C10—C11—C12—C13 | 0.8 (4) |
| C1—O1—C5—C4 | −4.8 (4) | C11—C12—C13—O3 | 180.0 (2) |
| C1—O1—C5—C6 | 176.1 (3) | C11—C12—C13—C14 | −1.5 (4) |
| C3—C4—C5—O1 | 179.4 (2) | C16—O3—C13—C12 | −6.5 (4) |
| C3—C4—C5—C6 | −1.5 (3) | C16—O3—C13—C14 | 174.9 (2) |
| C2—O2—C6—C7 | −1.8 (4) | C17—O4—C14—C15 | −1.1 (3) |
| C2—O2—C6—C5 | 178.7 (2) | C17—O4—C14—C13 | 178.8 (2) |
| O1—C5—C6—O2 | 0.3 (3) | C12—C13—C14—C15 | 1.0 (3) |
| C4—C5—C6—O2 | −178.9 (2) | O3—C13—C14—C15 | 179.7 (2) |
| O1—C5—C6—C7 | −179.3 (2) | C12—C13—C14—O4 | −178.9 (2) |
| C4—C5—C6—C7 | 1.5 (3) | O3—C13—C14—O4 | −0.2 (3) |
| O2—C6—C7—C8 | −180.0 (2) | O4—C14—C15—C10 | −180.0 (2) |
| C5—C6—C7—C8 | −0.5 (3) | C13—C14—C15—C10 | 0.1 (3) |
| C4—C3—C8—C7 | 0.6 (3) | C11—C10—C15—C14 | −0.7 (3) |
| C4—C3—C8—C9 | −177.74 (19) | C9—C10—C15—C14 | −177.8 (2) |
| C6—C7—C8—C3 | −0.6 (3) | C10—C9—C18—C19 | 72.2 (3) |
| C6—C7—C8—C9 | 177.71 (19) | C8—C9—C18—C19 | −158.8 (2) |
| C3—C8—C9—C10 | −147.48 (19) | C10—C9—C18—C22 | −108.8 (2) |
| C7—C8—C9—C10 | 34.3 (3) | C8—C9—C18—C22 | 20.2 (3) |
| C3—C8—C9—C18 | 83.7 (2) | C22—C18—C19—C20 | −0.9 (4) |
| C7—C8—C9—C18 | −94.6 (2) | C9—C18—C19—C20 | 178.2 (3) |
| C8—C9—C10—C11 | −97.2 (3) | C21—N1—C20—C19 | 0.5 (4) |
| C18—C9—C10—C11 | 31.7 (3) | C18—C19—C20—N1 | 0.4 (5) |
| C8—C9—C10—C15 | 79.6 (2) | C20—N1—C21—C22 | −0.9 (4) |
| C18—C9—C10—C15 | −151.5 (2) | C19—C18—C22—C21 | 0.5 (3) |
| C15—C10—C11—C12 | 0.2 (4) | C9—C18—C22—C21 | −178.5 (2) |
| C9—C10—C11—C12 | 177.1 (2) | N1—C21—C22—C18 | 0.4 (4) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O5—H5···N1 | 0.82 | 2.04 | 2.842 (4) | 167 |