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# (Pyridine-2,6-diyldimethylene)bis(diphenylmethanol)

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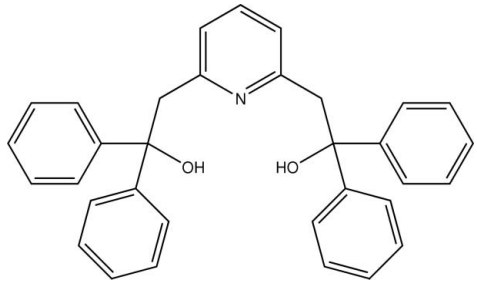
Key indicators: single-crystal X-ray study; $T$ = 291 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.054; $wR$ factor = 0.127; data-to-parameter ratio = 8.9.

In the title compound, $C_{33}H_{29}NO_2$, the central pyridyl ring makes dihedral angles of 42.71 (16), 44.78 (16), 85.47 (12) and 76.74 (12)° with the four phenyl rings. There are two intramolecular O—H···N hydrogen bonds. In the crystal structure, molecules are linked into a chain running along the $b$ axis by a weak C—H···$\pi$ interaction.

## Related literature

For organometallic pincer complexes, see: Dupont *et al.* (2005); Gauvin *et al.* (2001); Haenel *et al.* (2001); van der Boom & Milstein (2003); van der Boom *et al.* (1997); Vigalok & Milstein (2001); Bergbreiter *et al.* (1999). The title compound was prepared according to the procedure described by Berg & Holm (1985).




## Experimental

### Crystal data

$C_{33}H_{29}NO_2$
$M_r$ = 471.57
Monoclinic, $Cc$
$a$ = 18.492 (3) Å
$b$ = 10.1039 (17) Å
$c$ = 16.097 (3) Å
$\beta$ = 121.234 (2)°
$V$ = 2571.7 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 291 (2) K
0.30 × 0.26 × 0.24 mm

### Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min}$ = 0.980, $T_{max}$ = 0.982
10905 measured reflections
2960 independent reflections
2695 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.040

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.127
$S$ = 1.04
2960 reflections
331 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···N1 | 0.82 (5) | 2.34 (5) | 3.013 (4) | 139 (4) |
| O2—H2$A$···N1 | 0.82 (5) | 2.20 (5) | 2.854 (4) | 136 (4) |
| C31—H31···$Cg1^{i}$ | 0.93 | 3.08 | 3.973 (3) | 162 |

Symmetry code: (i) $x, y-1, z$. $Cg1$ is the centroid of the C8–C13 ring.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2375).

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# supplementary materials

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# (Pyridine-2,6-diyldimethylene)bis(diphenylmethanol)

**W.-J. Gu and B.-X. Wang**

## Comment

Currently, organometallic pincer complexes attract much attention because of their widespread applications in catalysis and material sciences (Dupont *et al.*, 2005; van der Boom & Milstein, 2003). Major recent findings have been the generation of efficient dehydrogenation (Haenel *et al.*, 2001) and Heck type catalysts (Bergbreiter *et al.*, 1999), activation of strong C—O (van der Boom *et al.*, 1997) and C—C bonds (Gauvin *et al.*, 2001), and trapping of various intermediates and unusual molecules (Vigalok & Milstein, 2001). 2,6-Bis(2-hydroxy-2,2-diphenylethyl)pyridine, (I), could coordinate with transition metals to form pincer complexes. In our studies, we have got its single crystals and herein reported its crystal structure.

The crystal structure of title compound, $C_{33}H_{29}NO_2$, reveals that all the bond lengths and angles have normal values. Each asymmetric unit in (I) contains four phenyl rings A (C8—C13), B (C14—C19), C (C22—C27) D (C28—C33) and a pyridyl ring E (N1/C1—C5). The rings A, B, C, D and E are all not coplanar, their dihedral angles between rings A and B, B and E, E and C, C and D being 68.13 (15), 44.79 (16), 85.48 (11) and 86.85 (14)°, respectively. The dihedral angles between rings A and E, B and E, C and E, D and E are 42.71 (16), 44.78 (16), 85.47 (12) and 76.74 (12)°, respectively. In the molecule there are two intramolecular O—H···N hydrogen bonds (Table 1 and Fig. 1). In the crystal, there is a weak C—H···π interaction (C31—H31···*Cg*1$^{i}$, i: *x*, -1 + *y*, *z*; *Cg*1 is the centroid of ring A) between the neighbouring molecules (Table 1). Through the weak C—H···π interactions, the one-dimensional chains are formed along the *b* axis (Fig. 2).

## Experimental

2,6-Bis(2-hydroxy-2,2-diphenylethyl)pyridine was prepared by 2,6-lutidine and benzophenone (yield 30%) according to a procedure described in the literature (Berg & Holm, 1985). Colorless crystals were obtained by recrystallized from light petroleum-ethyl acetate (*v*/*v* 5/1) at room temperature.

$^1$H-NMR ($CDCl_3$, 400 MHz) δ: 7.17–7.37 (21 H, m, 4Ph + 4-H), 6.69 (2 H, d, J = 7.5 Hz, 3-H + 5-H), 5.25 (2 H, s, 2OH), 3.68 (4 H, s, 2$CH_2$).

## Refinement

H atoms bonded to O atoms were located in a difference map and their positional parameters were refined with $U_{iso}$(H) = 1.2$U_{eq}$(O). Other H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined using a riding model, with $U_{iso}$(H) = 1.2$U_{eq}$(C). In the absence of significant anomalous scattering effects, Friedel pairs have been merged.

**Figures**

Fig. 1. A view of the title compound showing the atom-numbering scheme and displacement ellipsoids drawn at 30% probability level. Dashed lines indicate hydrogen bonds and all H atoms except those involved in hydrogen bonding have been omitted for clarify.

Fig. 2. The 1-D chain, viewed along the *a* axis. Dashed lines indicate the C—H···π interaction between the neighbouring molecules [symmetry code: (i) *x*, -1 + *y*, *z*]. H atoms not involved in the interaction have been omitted for clarify.

**(Pyridine-2,6-diyldimethylene)bis(diphenylmethanol)**

*Crystal data*

| | |
|---|---|
| $C_{33}H_{29}NO_2$ | $F_{000}$ = 1000 |
| $M_r$ = 471.57 | $D_x$ = 1.218 Mg m$^{-3}$ |
| Monoclinic, *Cc* | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: C -2yc | Cell parameters from 6093 reflections |
| *a* = 18.492 (3) Å | θ = 2.4–27.5º |
| *b* = 10.1039 (17) Å | μ = 0.08 mm$^{-1}$ |
| *c* = 16.097 (3) Å | *T* = 291 (2) K |
| β = 121.234 (2)º | Block, colourless |
| *V* = 2571.7 (8) Å$^3$ | 0.30 × 0.26 × 0.24 mm |
| *Z* = 4 | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX CCD diffractometer | 2960 independent reflections |
| Radiation source: sealed tube | 2695 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.040 |
| *T* = 291(2) K | $\theta_{max}$ = 27.6º |
| φ and ω scans | $\theta_{min}$ = 2.4º |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2000) | *h* = −24→21 |
| $T_{min}$ = 0.980, $T_{max}$ = 0.982 | *k* = −13→13 |
| 10905 measured reflections | *l* = −20→20 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.054 | H atoms treated by a mixture of independent and constrained refinement |

| | |
|---|---|
| $wR(F^2) = 0.127$ | $w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 1.99P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.04$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 2960 reflections | $\Delta\rho_{max} = 0.41$ e Å$^{-3}$ |
| 331 parameters | $\Delta\rho_{min} = -0.39$ e Å$^{-3}$ |
| 2 restraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*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.

Least-squares planes (*x*,*y*,*z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)

-2.3376 (0.0303) *x* - 5.1684 (0.0138) *y* + 12.7530 (0.0167) *z* = 0.3615 (0.0248)

* 0.0017 (0.0025) C8 * -0.0041 (0.0026) C9 * 0.0000 (0.0028) C10 * 0.0065 (0.0029) C11 * -0.0089 (0.0030) C12 * 0.0048 (0.0028) C13

Rms deviation of fitted atoms = 0.0052

15.5569 (0.0164) *x* - 0.4094 (0.0178) *y* + 0.3974 (0.0262) *z* = 7.3929 (0.0188)

Angle to previous plane (with approximate e.s.d.) = 68.13 (0.15)

* -0.0126 (0.0027) C14 * 0.0109 (0.0029) C15 * -0.0002 (0.0031) C16 * -0.0092 (0.0030) C17 * 0.0076 (0.0030) C18 * 0.0035 (0.0029) C19

Rms deviation of fitted atoms = 0.0085

- 8.9504 (0.0218) *x* + 7.2017 (0.0078) *y* - 2.9469 (0.0230) *z* = 2.8842 (0.0118)

Angle to previous plane (with approximate e.s.d.) = 44.79 (0.16)

* 0.0048 (0.0024) N1 * 0.0001 (0.0031) C1 * 0.0004 (0.0030) C2 * -0.0045 (0.0028) C3 * 0.0087 (0.0031) C4 * -0.0091 (0.0024) C5 * -0.0002 (0.0022) C6

Rms deviation of fitted atoms = 0.0054

16.3880 (0.0176) *x* + 4.6610 (0.0180) *y* - 7.9763 (0.0254) *z* = 6.8874 (0.0075)

Angle to previous plane (with approximate e.s.d.) = 85.48 (0.11)

* -0.0149 (0.0028) C22 * 0.0006 (0.0031) C23 * 0.0132 (0.0033) C24 * -0.0125 (0.0034) C25 * -0.0023 (0.0036) C26 * 0.0160 (0.0032) C27

Rms deviation of fitted atoms = 0.0117

- 6.3202 (0.0322) *x* + 6.0939 (0.0141) *y* + 12.7724 (0.0177) *z* = 5.2974 (0.0138)

Angle to previous plane (with approximate e.s.d.) = 86.85 (0.14)

* 0.0000 (0.0028) C28 * 0.0000 (0.0032) C29 * 0.0000 (0.0033) C30 * 0.0000 (0.0031) C31 * 0.0000 (0.0031) C32 * 0.0000 (0.0029) C33

Rms deviation of fitted atoms = 0.0000

##########

Least-squares planes (*x*,*y*,*z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)

- 8.9516 (0.0242) *x* + 7.2008 (0.0111) *y* - 2.9471 (0.0232) *z* = 2.8832 (0.0159)

* 0.0046 (0.0022) N1 * -0.0001 (0.0025) C1 * 0.0003 (0.0027) C2 * -0.0045 (0.0028) C3 * 0.0088 (0.0028) C4 * -0.0091 (0.0025) C5

Rms deviation of fitted atoms = 0.0058

- 2.3376 (0.0303) *x* - 5.1684 (0.0138) *y* + 12.7530 (0.0167) *z* = 0.3615 (0.0248)

Angle to previous plane (with approximate e.s.d.) = 42.71 (0.16)

* 0.0017 (0.0025) C8 * -0.0041 (0.0026) C9 * 0.0000 (0.0028) C10 * 0.0065 (0.0029) C11 * -0.0089 (0.0030) C12 * 0.0048 (0.0028) C13

Rms deviation of fitted atoms = 0.0052

- 8.9516 (0.0242) *x* + 7.2008 (0.0111) *y* - 2.9471 (0.0232) *z* = 2.8832 (0.0159)

Angle to previous plane (with approximate e.s.d.) = 42.71 (0.16)

* 0.0046 (0.0022) N1 * -0.0001 (0.0025) C1 * 0.0003 (0.0027) C2 * -0.0045 (0.0028) C3 * 0.0088 (0.0028) C4 * -0.0091 (0.0025) C5

Rms deviation of fitted atoms = 0.0058

15.5569 (0.0164) *x* - 0.4094 (0.0178) *y* + 0.3974 (0.0262) *z* = 7.3929 (0.0188)

Angle to previous plane (with approximate e.s.d.) = 44.78 (0.16)

* -0.0126 (0.0027) C14 * 0.0109 (0.0029) C15 * -0.0002 (0.0031) C16 * -0.0092 (0.0030) C17 * 0.0076 (0.0030) C18 * 0.0035 (0.0029) C19

Rms deviation of fitted atoms = 0.0085

- 8.9516 (0.0242) *x* + 7.2008 (0.0111) *y* - 2.9471 (0.0232) *z* = 2.8832 (0.0159)

Angle to previous plane (with approximate e.s.d.) = 44.78 (0.16)

* 0.0046 (0.0022) N1 * -0.0001 (0.0025) C1 * 0.0003 (0.0027) C2 * -0.0045 (0.0028) C3 * 0.0088 (0.0028) C4 * -0.0091 (0.0025) C5

Rms deviation of fitted atoms = 0.0058

16.3880 (0.0176) *x* + 4.6610 (0.0180) *y* - 7.9763 (0.0254) *z* = 6.8874 (0.0075)

Angle to previous plane (with approximate e.s.d.) = 85.47 (0.12)

* -0.0149 (0.0028) C22 * 0.0006 (0.0031) C23 * 0.0132 (0.0033) C24 * -0.0125 (0.0034) C25 * -0.0023 (0.0036) C26 * 0.0160 (0.0032) C27

Rms deviation of fitted atoms = 0.0117

- 8.9516 (0.0242) *x* + 7.2008 (0.0111) *y* - 2.9471 (0.0232) *z* = 2.8832 (0.0159)

Angle to previous plane (with approximate e.s.d.) = 85.47 (0.12)

* 0.0046 (0.0022) N1 * -0.0001 (0.0025) C1 * 0.0003 (0.0027) C2 * -0.0045 (0.0028) C3 * 0.0088 (0.0028) C4 * -0.0091 (0.0025) C5

Rms deviation of fitted atoms = 0.0058

- 6.3202 (0.0322) *x* + 6.0939 (0.0141) *y* + 12.7724 (0.0177) *z* = 5.2974 (0.0138)

Angle to previous plane (with approximate e.s.d.) = 76.74 (0.12)

* 0.0000 (0.0028) C28 * 0.0000 (0.0032) C29 * 0.0000 (0.0033) C30 * 0.0000 (0.0031) C31 * 0.0000 (0.0031) C32 * 0.0000 (0.0029) C33

Rms deviation of fitted atoms = 0.0000

**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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.3187 (2) | 0.9740 (4) | 0.4335 (3) | 0.0489 (8) |
| C2 | 0.2728 (3) | 0.9343 (4) | 0.4758 (3) | 0.0537 (9) |
| H2 | 0.2837 | 0.9721 | 0.5339 | 0.064* |
| C3 | 0.2110 (3) | 0.8385 (4) | 0.4312 (3) | 0.0548 (10) |
| H3 | 0.1804 | 0.8102 | 0.4589 | 0.066* |
| C4 | 0.1957 (3) | 0.7858 (5) | 0.3442 (3) | 0.0563 (10) |
| H4 | 0.1535 | 0.7228 | 0.3115 | 0.068* |
| C5 | 0.2441 (2) | 0.8281 (4) | 0.3068 (2) | 0.0427 (7) |
| C6 | 0.3866 (3) | 1.0774 (4) | 0.4800 (3) | 0.0526 (9) |
| H6A | 0.3791 | 1.1265 | 0.5269 | 0.063* |
| H6B | 0.3803 | 1.1391 | 0.4305 | 0.063* |
| C7 | 0.4777 (2) | 1.0188 (3) | 0.5323 (2) | 0.0416 (7) |
| C8 | 0.5447 (2) | 1.1246 (4) | 0.5841 (2) | 0.0443 (8) |
| C9 | 0.5332 (3) | 1.2363 (4) | 0.6268 (3) | 0.0535 (9) |
| H9 | 0.4815 | 1.2498 | 0.6223 | 0.064* |
| C10 | 0.5985 (3) | 1.3284 (4) | 0.6764 (3) | 0.0532 (9) |
| H10 | 0.5901 | 1.4024 | 0.7047 | 0.064* |
| C11 | 0.6736 (3) | 1.3097 (4) | 0.6831 (3) | 0.0591 (11) |

| | | | | |
|---|---|---|---|---|
| H11 | 0.7171 | 1.3705 | 0.7169 | 0.071* |
| C12 | 0.6865 (3) | 1.2012 (4) | 0.6403 (3) | 0.0581 (11) |
| H12 | 0.7380 | 1.1906 | 0.6439 | 0.070* |
| C13 | 0.6231 (3) | 1.1077 (4) | 0.5919 (3) | 0.0542 (9) |
| H13 | 0.6327 | 1.0338 | 0.5645 | 0.065* |
| C14 | 0.4830 (2) | 0.9113 (4) | 0.6037 (3) | 0.0428 (7) |
| C15 | 0.4833 (3) | 0.9465 (4) | 0.6872 (3) | 0.0518 (9) |
| H15 | 0.4867 | 1.0353 | 0.7039 | 0.062* |
| C16 | 0.4785 (3) | 0.8508 (4) | 0.7464 (3) | 0.0547 (10) |
| H16 | 0.4777 | 0.8753 | 0.8016 | 0.066* |
| C17 | 0.4751 (3) | 0.7177 (4) | 0.7218 (3) | 0.0584 (11) |
| H17 | 0.4713 | 0.6525 | 0.7601 | 0.070* |
| C18 | 0.4773 (3) | 0.6844 (4) | 0.6424 (3) | 0.0603 (10) |
| H18 | 0.4763 | 0.5953 | 0.6275 | 0.072* |
| C19 | 0.4810 (3) | 0.7780 (4) | 0.5822 (3) | 0.0574 (10) |
| H19 | 0.4823 | 0.7518 | 0.5275 | 0.069* |
| C20 | 0.2248 (2) | 0.7717 (4) | 0.2092 (3) | 0.0482 (8) |
| H20A | 0.1883 | 0.8332 | 0.1582 | 0.058* |
| H20B | 0.1938 | 0.6895 | 0.1974 | 0.058* |
| C21 | 0.3036 (2) | 0.7445 (3) | 0.2015 (2) | 0.0392 (7) |
| C22 | 0.2752 (2) | 0.6821 (4) | 0.1024 (2) | 0.0410 (7) |
| C23 | 0.3043 (3) | 0.5634 (4) | 0.0909 (3) | 0.0542 (10) |
| H23 | 0.3442 | 0.5168 | 0.1454 | 0.065* |
| C24 | 0.2749 (3) | 0.5108 (5) | −0.0018 (3) | 0.0609 (11) |
| H24 | 0.2961 | 0.4306 | −0.0084 | 0.073* |
| C25 | 0.2159 (3) | 0.5761 (4) | −0.0816 (3) | 0.0561 (10) |
| H25 | 0.1949 | 0.5396 | −0.1430 | 0.067* |
| C26 | 0.1877 (3) | 0.6941 (5) | −0.0720 (3) | 0.0631 (12) |
| H26 | 0.1476 | 0.7396 | −0.1271 | 0.076* |
| C27 | 0.2175 (3) | 0.7490 (4) | 0.0192 (3) | 0.0602 (11) |
| H27 | 0.1985 | 0.8320 | 0.0243 | 0.072* |
| C28 | 0.3695 (2) | 0.6555 (3) | 0.2848 (2) | 0.0383 (7) |
| C29 | 0.4551 (2) | 0.6722 (4) | 0.3192 (3) | 0.0573 (10) |
| H29 | 0.4729 | 0.7406 | 0.2954 | 0.069* |
| C30 | 0.5141 (3) | 0.5866 (4) | 0.3892 (3) | 0.0618 (11) |
| H30 | 0.5714 | 0.5978 | 0.4123 | 0.074* |
| C31 | 0.4874 (3) | 0.4844 (4) | 0.4249 (3) | 0.0521 (10) |
| H31 | 0.5269 | 0.4271 | 0.4717 | 0.063* |
| C32 | 0.4018 (3) | 0.4677 (5) | 0.3905 (3) | 0.0618 (12) |
| H32 | 0.3840 | 0.3992 | 0.4143 | 0.074* |
| C33 | 0.3429 (2) | 0.5532 (4) | 0.3204 (3) | 0.0470 (9) |
| H33 | 0.2856 | 0.5421 | 0.2974 | 0.056* |
| N1 | 0.3029 (2) | 0.9203 (3) | 0.3487 (2) | 0.0467 (7) |
| O1 | 0.49165 (17) | 0.9616 (3) | 0.46054 (19) | 0.0497 (6) |
| H1 | 0.451 (3) | 0.917 (5) | 0.423 (4) | 0.060* |
| O2 | 0.34180 (18) | 0.8688 (3) | 0.2017 (2) | 0.0503 (6) |
| H2A | 0.352 (3) | 0.911 (5) | 0.250 (4) | 0.060* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.054 (2) | 0.0442 (19) | 0.0435 (19) | 0.0163 (16) | 0.0219 (17) | 0.0076 (15) |
| C2 | 0.052 (2) | 0.066 (2) | 0.048 (2) | 0.0145 (19) | 0.0301 (19) | −0.0020 (18) |
| C3 | 0.060 (2) | 0.059 (2) | 0.065 (2) | 0.0100 (19) | 0.046 (2) | 0.008 (2) |
| C4 | 0.052 (2) | 0.063 (2) | 0.056 (2) | −0.0030 (18) | 0.0294 (19) | 0.0000 (19) |
| C5 | 0.0350 (16) | 0.0516 (19) | 0.0351 (16) | 0.0133 (14) | 0.0136 (14) | 0.0095 (14) |
| C6 | 0.060 (2) | 0.0430 (19) | 0.044 (2) | 0.0092 (17) | 0.0187 (18) | 0.0022 (15) |
| C7 | 0.0529 (19) | 0.0390 (16) | 0.0342 (16) | 0.0037 (14) | 0.0235 (15) | −0.0013 (13) |
| C8 | 0.053 (2) | 0.0441 (18) | 0.0312 (16) | −0.0001 (15) | 0.0186 (15) | 0.0053 (14) |
| C9 | 0.061 (2) | 0.049 (2) | 0.0378 (19) | 0.0057 (17) | 0.0163 (17) | −0.0007 (15) |
| C10 | 0.066 (2) | 0.0429 (19) | 0.043 (2) | 0.0024 (17) | 0.0228 (18) | −0.0019 (15) |
| C11 | 0.058 (2) | 0.049 (2) | 0.053 (2) | −0.0167 (18) | 0.017 (2) | −0.0018 (18) |
| C12 | 0.070 (3) | 0.055 (2) | 0.057 (2) | −0.023 (2) | 0.038 (2) | −0.0020 (18) |
| C13 | 0.056 (2) | 0.050 (2) | 0.058 (2) | −0.0109 (17) | 0.030 (2) | −0.0053 (18) |
| C14 | 0.0409 (17) | 0.0477 (18) | 0.0437 (18) | 0.0091 (14) | 0.0248 (15) | 0.0041 (14) |
| C15 | 0.066 (2) | 0.054 (2) | 0.062 (2) | 0.0202 (18) | 0.052 (2) | 0.0165 (18) |
| C16 | 0.056 (2) | 0.069 (2) | 0.063 (2) | 0.0193 (19) | 0.047 (2) | 0.030 (2) |
| C17 | 0.059 (2) | 0.061 (2) | 0.059 (2) | −0.0047 (19) | 0.034 (2) | 0.035 (2) |
| C18 | 0.054 (2) | 0.054 (2) | 0.061 (3) | −0.0055 (19) | 0.021 (2) | 0.011 (2) |
| C19 | 0.061 (2) | 0.0410 (18) | 0.062 (2) | −0.0001 (17) | 0.026 (2) | 0.0063 (18) |
| C20 | 0.0379 (17) | 0.053 (2) | 0.0414 (19) | −0.0019 (15) | 0.0122 (15) | 0.0011 (16) |
| C21 | 0.0383 (16) | 0.0383 (16) | 0.0353 (16) | −0.0021 (13) | 0.0151 (14) | 0.0014 (13) |
| C22 | 0.0394 (17) | 0.0510 (19) | 0.0329 (16) | −0.0028 (14) | 0.0189 (14) | 0.0014 (14) |
| C23 | 0.049 (2) | 0.068 (3) | 0.0390 (19) | 0.0143 (19) | 0.0181 (17) | −0.0059 (17) |
| C24 | 0.056 (2) | 0.068 (3) | 0.057 (2) | 0.013 (2) | 0.028 (2) | −0.026 (2) |
| C25 | 0.064 (2) | 0.057 (2) | 0.048 (2) | 0.0025 (18) | 0.030 (2) | −0.0255 (17) |
| C26 | 0.060 (2) | 0.072 (3) | 0.043 (2) | 0.036 (2) | 0.0165 (19) | 0.003 (2) |
| C27 | 0.062 (2) | 0.056 (2) | 0.043 (2) | −0.0024 (19) | 0.0134 (19) | 0.0019 (17) |
| C28 | 0.0431 (17) | 0.0413 (16) | 0.0285 (14) | −0.0007 (13) | 0.0172 (13) | 0.0062 (12) |
| C29 | 0.0421 (19) | 0.061 (2) | 0.052 (2) | −0.0080 (17) | 0.0125 (17) | 0.0114 (18) |
| C30 | 0.0364 (19) | 0.064 (2) | 0.067 (3) | −0.0021 (17) | 0.0139 (19) | 0.005 (2) |
| C31 | 0.060 (2) | 0.057 (2) | 0.0392 (17) | 0.0355 (18) | 0.0257 (17) | 0.0174 (16) |
| C32 | 0.069 (3) | 0.072 (3) | 0.061 (2) | 0.037 (2) | 0.046 (2) | 0.041 (2) |
| C33 | 0.0476 (18) | 0.060 (2) | 0.057 (2) | 0.0232 (16) | 0.0431 (18) | 0.0297 (18) |
| N1 | 0.0441 (16) | 0.0486 (17) | 0.0441 (16) | 0.0099 (13) | 0.0205 (13) | 0.0102 (13) |
| O1 | 0.0531 (15) | 0.0579 (16) | 0.0533 (15) | −0.0102 (12) | 0.0382 (13) | −0.0174 (13) |
| O2 | 0.0600 (16) | 0.0400 (13) | 0.0445 (14) | −0.0098 (12) | 0.0226 (13) | 0.0057 (11) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.353 (5) | C17—H17 | 0.9300 |
| C1—C2 | 1.394 (6) | C18—C19 | 1.382 (6) |
| C1—C6 | 1.501 (6) | C18—H18 | 0.9300 |
| C2—C3 | 1.381 (6) | C19—H19 | 0.9300 |
| C2—H2 | 0.9300 | C20—C21 | 1.549 (5) |
| C3—C4 | 1.384 (6) | C20—H20A | 0.9700 |

| | | | |
|---|---|---|---|
| C3—H3 | 0.9300 | C20—H20B | 0.9700 |
| C4—C5 | 1.381 (5) | C21—O2 | 1.440 (4) |
| C4—H4 | 0.9300 | C21—C22 | 1.533 (5) |
| C5—N1 | 1.321 (5) | C21—C28 | 1.548 (5) |
| C5—C20 | 1.530 (5) | C22—C23 | 1.366 (5) |
| C6—C7 | 1.557 (5) | C22—C27 | 1.381 (5) |
| C6—H6A | 0.9700 | C23—C24 | 1.401 (5) |
| C6—H6B | 0.9700 | C23—H23 | 0.9300 |
| C7—O1 | 1.431 (4) | C24—C25 | 1.351 (6) |
| C7—C8 | 1.518 (5) | C24—H24 | 0.9300 |
| C7—C14 | 1.547 (5) | C25—C26 | 1.342 (5) |
| C8—C9 | 1.394 (5) | C25—H25 | 0.9300 |
| C8—C13 | 1.399 (6) | C26—C27 | 1.388 (6) |
| C9—C10 | 1.401 (6) | C26—H26 | 0.9300 |
| C9—H9 | 0.9300 | C27—H27 | 0.9300 |
| C10—C11 | 1.350 (6) | C28—C29 | 1.390 (5) |
| C10—H10 | 0.9300 | C28—C33 | 1.390 (4) |
| C11—C12 | 1.380 (6) | C29—C30 | 1.390 (6) |
| C11—H11 | 0.9300 | C29—H29 | 0.9300 |
| C12—C13 | 1.388 (5) | C30—C31 | 1.390 (6) |
| C12—H12 | 0.9300 | C30—H30 | 0.9300 |
| C13—H13 | 0.9300 | C31—C32 | 1.390 (6) |
| C14—C19 | 1.387 (5) | C31—H31 | 0.9300 |
| C14—C15 | 1.387 (5) | C32—C33 | 1.390 (5) |
| C15—C16 | 1.392 (5) | C32—H32 | 0.9300 |
| C15—H15 | 0.9300 | C33—H33 | 0.9300 |
| C16—C17 | 1.394 (6) | O1—H1 | 0.82 (5) |
| C16—H16 | 0.9300 | O2—H2A | 0.82 (5) |
| C17—C18 | 1.342 (7) | | |
| N1—C1—C2 | 120.6 (4) | C17—C18—C19 | 122.3 (4) |
| N1—C1—C6 | 118.0 (4) | C17—C18—H18 | 118.9 |
| C2—C1—C6 | 121.4 (4) | C19—C18—H18 | 118.9 |
| C3—C2—C1 | 119.8 (4) | C18—C19—C14 | 119.5 (4) |
| C3—C2—H2 | 120.1 | C18—C19—H19 | 120.2 |
| C1—C2—H2 | 120.1 | C14—C19—H19 | 120.2 |
| C2—C3—C4 | 118.4 (4) | C5—C20—C21 | 114.8 (3) |
| C2—C3—H3 | 120.8 | C5—C20—H20A | 108.6 |
| C4—C3—H3 | 120.8 | C21—C20—H20A | 108.6 |
| C5—C4—C3 | 118.9 (4) | C5—C20—H20B | 108.6 |
| C5—C4—H4 | 120.5 | C21—C20—H20B | 108.6 |
| C3—C4—H4 | 120.5 | H20A—C20—H20B | 107.5 |
| N1—C5—C4 | 122.9 (4) | O2—C21—C22 | 105.3 (3) |
| N1—C5—C20 | 118.7 (3) | O2—C21—C28 | 109.9 (3) |
| C4—C5—C20 | 118.3 (4) | C22—C21—C28 | 110.7 (3) |
| C1—C6—C7 | 113.3 (3) | O2—C21—C20 | 109.0 (3) |
| C1—C6—H6A | 108.9 | C22—C21—C20 | 109.0 (3) |
| C7—C6—H6A | 108.9 | C28—C21—C20 | 112.6 (3) |
| C1—C6—H6B | 108.9 | C23—C22—C27 | 117.3 (4) |
| C7—C6—H6B | 108.9 | C23—C22—C21 | 123.8 (3) |

| | | | |
|---|---|---|---|
| H6A—C6—H6B | 107.7 | C27—C22—C21 | 119.0 (3) |
| O1—C7—C8 | 106.9 (3) | C22—C23—C24 | 121.0 (4) |
| O1—C7—C14 | 110.4 (3) | C22—C23—H23 | 119.5 |
| C8—C7—C14 | 111.6 (3) | C24—C23—H23 | 119.5 |
| O1—C7—C6 | 108.3 (3) | C25—C24—C23 | 120.3 (4) |
| C8—C7—C6 | 112.1 (3) | C25—C24—H24 | 119.9 |
| C14—C7—C6 | 107.6 (3) | C23—C24—H24 | 119.9 |
| C9—C8—C13 | 118.1 (4) | C26—C25—C24 | 119.7 (4) |
| C9—C8—C7 | 123.4 (3) | C26—C25—H25 | 120.2 |
| C13—C8—C7 | 118.4 (3) | C24—C25—H25 | 120.2 |
| C8—C9—C10 | 120.8 (4) | C25—C26—C27 | 120.8 (4) |
| C8—C9—H9 | 119.6 | C25—C26—H26 | 119.6 |
| C10—C9—H9 | 119.6 | C27—C26—H26 | 119.6 |
| C11—C10—C9 | 120.0 (4) | C22—C27—C26 | 120.9 (4) |
| C11—C10—H10 | 120.0 | C22—C27—H27 | 119.5 |
| C9—C10—H10 | 120.0 | C26—C27—H27 | 119.5 |
| C10—C11—C12 | 120.5 (4) | C29—C28—C33 | 120.0 (3) |
| C10—C11—H11 | 119.7 | C29—C28—C21 | 119.8 (3) |
| C12—C11—H11 | 119.7 | C33—C28—C21 | 120.1 (3) |
| C11—C12—C13 | 120.5 (4) | C28—C29—C30 | 120.0 (4) |
| C11—C12—H12 | 119.7 | C28—C29—H29 | 120.0 |
| C13—C12—H12 | 119.7 | C30—C29—H29 | 120.0 |
| C12—C13—C8 | 120.0 (4) | C31—C30—C29 | 120.0 (4) |
| C12—C13—H13 | 120.0 | C31—C30—H30 | 120.0 |
| C8—C13—H13 | 120.0 | C29—C30—H30 | 120.0 |
| C19—C14—C15 | 118.6 (4) | C30—C31—C32 | 120.0 (3) |
| C19—C14—C7 | 120.8 (3) | C30—C31—H31 | 120.0 |
| C15—C14—C7 | 120.5 (3) | C32—C31—H31 | 120.0 |
| C14—C15—C16 | 121.0 (4) | C33—C32—C31 | 120.0 (4) |
| C14—C15—H15 | 119.5 | C33—C32—H32 | 120.0 |
| C16—C15—H15 | 119.5 | C31—C32—H32 | 120.0 |
| C15—C16—C17 | 119.0 (4) | C32—C33—C28 | 120.0 (3) |
| C15—C16—H16 | 120.5 | C32—C33—H33 | 120.0 |
| C17—C16—H16 | 120.5 | C28—C33—H33 | 120.0 |
| C18—C17—C16 | 119.6 (3) | C5—N1—C1 | 119.2 (3) |
| C18—C17—H17 | 120.2 | C7—O1—H1 | 109 (3) |
| C16—C17—H17 | 120.2 | C21—O2—H2A | 109 (3) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···N1 | 0.82 (5) | 2.34 (5) | 3.013 (4) | 139 (4) |
| O2—H2A···N1 | 0.82 (5) | 2.20 (5) | 2.854 (4) | 136 (4) |
| C31—H31···Cg1$^{i}$ | 0.93 | 3.08 | 3.973 (3) | 162 |

Symmetry codes: (i) *x*, *y*−1, *z*.

**Fig. 1**

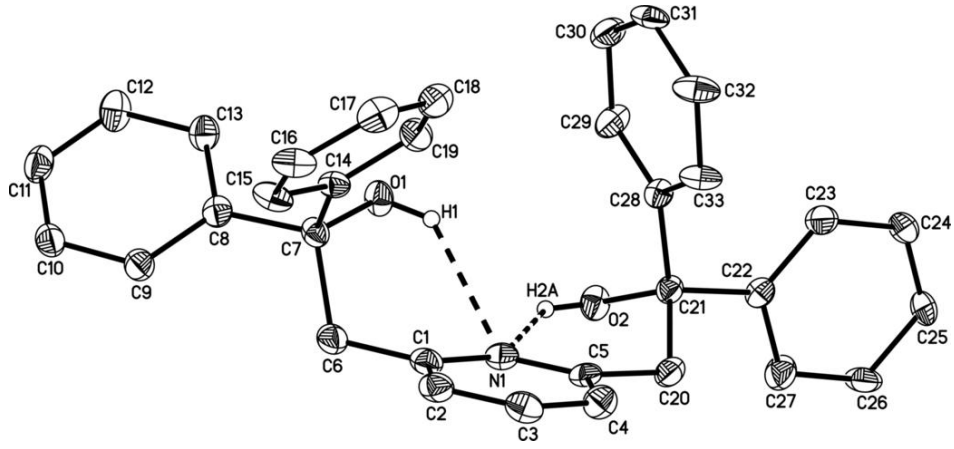
C31
C30
C32
C29
C12
C13
C17
C18
C16
C19
C14
C15
O1
C28
C33
C23
C24
C11
H1
C8
C7
C22
C10
C9
H2A
O2
C21
C1
C5
C25
C6
N1
C20
C27
C26
C2
C3
C4

**Fig. 2**