IUCrData

ISSN 2414-3146

# 2-({[(2*S*)-1-Hydroxy-1,1,3-triphenylpropan-2-yl]-imino}methyl)-4,6-bis(4-methylphenyl)phenol

**Veronica L. Show, Emily Y. Fok and Adam R. Johnson***

Department of Chemistry, Harvey Mudd College, 301 Platt Blvd., Claremont, CA 91711, USA. *Correspondence e-mail: adam_johnson@hmc.edu

Received 9 November 2020
Accepted 1 December 2020

Edited by J. Simpson, University of Otago, New Zealand

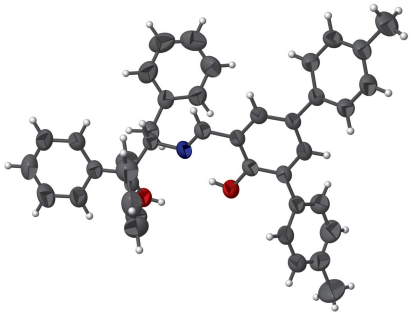
**Keywords:** crystal structure; Schiff base; tridentate ligand; chiral.

**CCDC reference**: 2042781

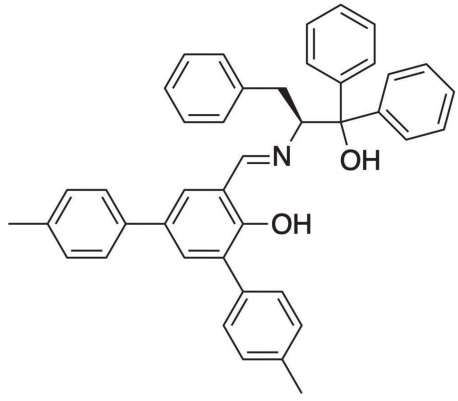
**Structural data**: full structural data are available from iucrdata.iucr.org

The title compound, $C_{42}H_{37}NO_2$, crystallizes in the orthorhombic space group $P2_12_12_1$ with one molecule in the asymmetric unit. An intramolecular hydrogen bond orients the phenol hydroxyl group toward the imine nitrogen. The aliphatic alcohol is engaged in a weak intramolecular hydrogen bond with the imine nitrogen.

**3D view**



**Chemical scheme**




## Structure description

We have synthesized a number of chiral imine diols by Schiff-base condensation of the corresponding salicylaldehydes with (*S*)-2-amino-1,1,3-triphenylpropanol (Kang *et al.*, 2004; Liu *et al.*, 2004). These compounds serve as ligands for titanium for the asymmetric intramolecular hydroamination of aminoallenes (Sha *et al.*, 2019; Fok *et al.*, 2020). The absolute structure parameter of 0.1 (7) has a large uncertainty but the absolute configuration was verified by synthesis and polarimetry.

The compound reported here has the expected imine–phenol structure (Fig. 1) as opposed to the iminium–phenoxide tautomer seen in derivatives with less steric bulk. There is an intramolecular O2—H2···N1 hydrogen bond, Table 1. The phenol aromatic ring (C23–C28) is essentially co-planar with O2, C22, and N1; O2 is 0.046 (3) Å above the plane, C22 is 0.083 (4) Å above the plane, and N1 is 0.180 (3) Å above the plane. The C22—N1—C2—C1 torsion angle is 136.5 (3)°, and the N1—C2—C1—O1 torsion angle is 60.2 (3)°, and these result in the positioning of C1 and H1 being pointed towards the open space near N1, and a intramolecular O1—H1···N1 hydrogen bond. O1 becomes almost coplanar with the phenol ring, only 0.198 (3) Å below the plane.

Unlike the significant bond alternation seen in related structures (Sha *et al.*, 2019), the bonds within the phenol ring (C23–C28) are all between 1.391 (4) and 1.406 (4) Å. The aromatic rings on the benzyl and phenyl substituents have typical aromatic bond distances ranging from 1.34–1.41 Å. The aromatic C24—O2 bond at 1.354 (4) Å is

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2···N1 | 0.82 | 1.80 | 2.539 (4) | 148 |
| O1—H1···N1 | 0.82 | 2.46 | 2.763 (3) | 103 |

substantially shorter than the aliphatic C1—O1 bond of 1.435 (4) Å, as seen in related structures.

## Synthesis and crystallization

Details of the preparation of the title compound are shown in Fig. 2. 2-Hydroxy-3,5-di-4-(methyl)phenylbenzaldehyde (0.8654 g, 2.86 mmol) and (*S*)-2-amino-1,1,3-triphenylpropanol (0.8651 g, 2.86 mmol, 1 equiv.) were dissolved in ethanol (50 ml) and heated overnight at reflux. The solvent was removed *in vacuo*. The crude product was purified by flash column chromatography to yield an orange solid (1.348 g, 1.52 mmol, 80.1%). X-ray quality crystals were obtained by slow evaporation of a toluene solution. M.p. 107.7–108.4°C. $[\alpha]_D$: −165° ($c$ = 0.006 g ml$^{-1}$, EtOAc). Analysis calculated for $C_{42}H_{37}NO_2$: C, 85.83; H, 6.34; N, 2.38. Found: C, 85.47; H, 6.43; N, 2.35. $^1$H NMR (400 MHz, $CDCl_3$): 13.22 (*s*, 1H, ArO*H*), 7.72–6.99 (*m*, 26H, Ar*H*, *H*C=N), 4.41 (*dd*, 1H, $J$ = 10.0, 1.6 Hz, C*H*CH$_a$H$_b$Ph), 3.02 (apparent *d*, 1H, $J$ = 12.5 Hz, CHC*H*$_a$H$_b$Ph), 2.96 (*s*, 1H, O*H*), 2.89 (*dd*, 1H, $J$ = 13.8, 10.2 Hz, CHCH$_a$*H*$_b$Ph), 2.41 (*s*, 3H, CH$_3$), 2.38 (*s*, 3H, CH$_3$). $^{13}$C NMR (100 MHz, $CDCl_3$): 167.11 (H*C*=N), 157.34 (4°), 145.66 (4°), 144.20 (4°), 139.06 (4°), 137.42 (4°), 137.23 (4°), 136.76 (4°), 134.67 (4°), 132.07 (4°), 131.95 (CH), 130.03 (4°), 129.88 (CH), 129.64 (CH), 129.34 (CH), 129.18 (CH), 129.10 (CH), 128.64 (CH), 128.56 (CH), 128.51 (CH), 128.37 (CH), 127.18 (CH), 127.06 (CH), 126.53 (CH), 126.20 (CH), 125.97 (CH), 118.74 (4°), 79.84 (4°), 78.97 (CH, chiral center), 37.54 (CH$_2$), 21.37 (CH$_3$), 21.18 (CH$_3$). MS (APCI): $m/z$ 589 [$M$+H]$^+$. IR (ATR, diamond): (C═N) = 1628 cm$^{-1}$.

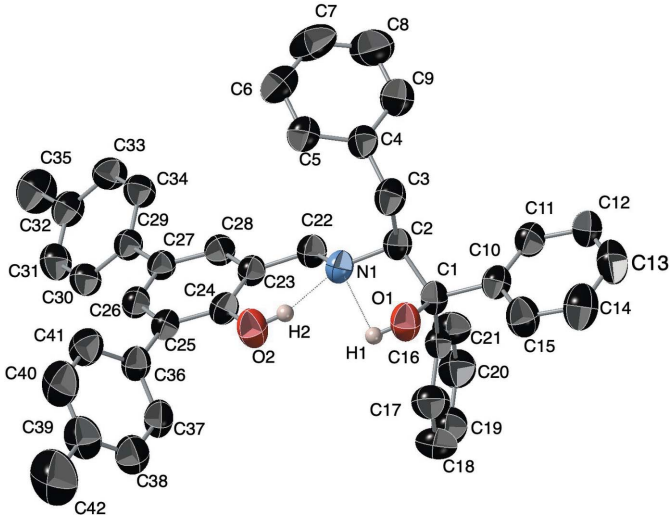



**Figure 1**
The asymmetric unit of the title compound, showing the atom-numbering scheme. The displacement ellipsoids are shown at the 50% probability level. Hydrogen atoms apart from H1 and H2 have been omitted for clarity and intramolecular hydrogen bonds are shown as dashed lines. Figure generated using *CrystalMaker* (Palmer, 2020).

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{42}H_{37}NO_2$ |
| $M_r$ | 587.72 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ |
| Temperature (K) | 293 |
| $a$, $b$, $c$ (Å) | 9.2554 (3), 11.5721 (4), 31.9214 (10) |
| $V$ (Å$^3$) | 3418.92 (19) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.07 |
| Crystal size (mm) | 0.31 × 0.16 × 0.11 |
| Data collection | |
| Diffractometer | XtaLAB Mini II |
| Absorption correction | Analytical [*CrysAlis PRO* (Rigaku OD, 2019) and ABSPACK (Rigaku OD, 2017)] |
| $T_{min}$, $T_{max}$ | 0.982, 0.994 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 88879, 6086, 3835 |
| $R_{int}$ | 0.074 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.597 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.052, 0.112, 1.01 |
| No. of reflections | 6086 |
| No. of parameters | 410 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.12, −0.11 |
| Absolute structure | Flack $x$ determined using 1229 quotients [$(I^+)$-$(I^-)$]/[$(I^+)$+$(I^-)$] (Parsons, *et al*, 2013) |
| Absolute structure parameter | 0.1 (7) |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2019), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009) and *CrystalMaker* (Palmer, 2020).

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

## Acknowledgements

The authors acknowledge technical assistance from Eric W. Reinheimer.

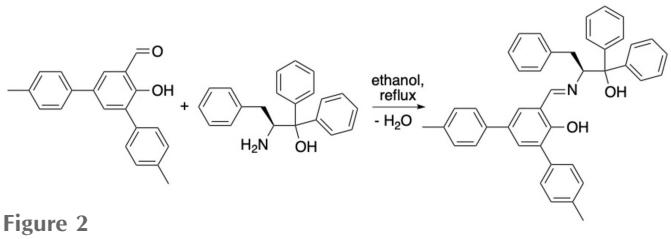



**Figure 2**
Synthesis of the title compound.

## References


Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Fok, E. Y., Show, V. L. & Johnson, A. R. (2020). *Polyhedron*. Submitted.

Kang, Y.-F., Liu, L., Wang, R., Yan, W.-J. & Zhou, Y.-F. (2004). *Tetrahedron Asymmetry*, **15**, 3155–3159.

Liu, L., Kang, Y.-F., Wang, R., Zhou, Y.-F., Chen, C., Ni, M. & Gong, M.-Z. (2004). *Tetrahedron Asymmetry*, **15**, 3757–3761.

Palmer, D. (2020). *CrystalMaker*. CrystalMaker Software, Bicester, England.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Rigaku OD (2017). *SCALE3 ABSPACK*. Rigaku Oxford Diffraction, Yarnton, England.

Rigaku OD (2019). *CrysAlis PRO*.. Rigaku Oxford Diffraction, Yarnton, England.

Sha, F., Mitchell, B. S., Ye, C. Z., Abelson, C. S., Reinheimer, E. W., LeMagueres, P., Ferrara, J. D., Takase, M. K. & Johnson, A. R. (2019). *Dalton Trans.* **48**, 9603–9616.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

# full crystallographic data

*IUCrData* (2020). **5**, x201576 [https://doi.org/10.1107/S241431462001576X]

## 2-({[(2*S*)-1-Hydroxy-1,1,3-triphenylpropan-2-yl]imino}methyl)-4,6-bis(4-methylphenyl)phenol

**Veronica L. Show, Emily Y. Fok and Adam R. Johnson**

**2-({[(2*S*)-1-Hydroxy-1,1,3-triphenylpropan-2-yl]imino}methyl)-4,6-bis(4-methylphenyl)phenol**

*Crystal data*

$C_{42}H_{37}NO_2$
$M_r$ = 587.72
Orthorhombic, $P2_12_12_1$
$a$ = 9.2554 (3) Å
$b$ = 11.5721 (4) Å
$c$ = 31.9214 (10) Å
$V$ = 3418.92 (19) Å$^3$
$Z$ = 4
$F(000)$ = 1248
$D_x$ = 1.142 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 14764 reflections
$\theta$ = 1.9–20.8°
$\mu$ = 0.07 mm$^{-1}$
$T$ = 293 K
Block, clear light yellow
0.31 × 0.16 × 0.11 mm

*Data collection*

XtaLAB Mini II diffractometer
Radiation source: fine-focus sealed X-ray tube, Rigaku (Mo) X-ray Source
Graphite monochromator
Detector resolution: 10.0000 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: analytical [CrysAlisPro (Rigaku OD, 2019) and ABSPACK (Rigaku OD, 2017)]
$T_{min}$ = 0.982, $T_{max}$ = 0.994
88879 measured reflections
6086 independent reflections
3835 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.074
$\theta_{max}$ = 25.1°, $\theta_{min}$ = 2.2°
$h$ = −11→11
$k$ = −13→13
$l$ = −38→38

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.112
$S$ = 1.01
6086 reflections
410 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 0.0355P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.11 e Å$^{-3}$
Absolute structure: Flack $x$ determined using 1229 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons, *et al*, 2013)
Absolute structure parameter: 0.1 (7)

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.6294 (3) | 0.4955 (2) | 0.24453 (7) | 0.0653 (7) |
| H1 | 0.689147 | 0.504113 | 0.263236 | 0.098* |
| O2 | 0.7917 (3) | 0.4887 (2) | 0.35057 (7) | 0.0692 (7) |
| H2 | 0.736655 | 0.464313 | 0.332477 | 0.104* |
| N1 | 0.6319 (3) | 0.3457 (2) | 0.31194 (8) | 0.0532 (7) |
| C1 | 0.6062 (4) | 0.3744 (3) | 0.23755 (10) | 0.0517 (9) |
| C2 | 0.5320 (4) | 0.3249 (3) | 0.27742 (9) | 0.0504 (9) |
| H2A | 0.516244 | 0.241633 | 0.274077 | 0.060* |
| C3 | 0.3884 (4) | 0.3847 (3) | 0.28819 (10) | 0.0639 (10) |
| H3A | 0.407654 | 0.463637 | 0.296750 | 0.077* |
| H3B | 0.328159 | 0.387332 | 0.263330 | 0.077* |
| C4 | 0.3080 (4) | 0.3237 (3) | 0.32255 (11) | 0.0556 (9) |
| C5 | 0.3318 (5) | 0.3464 (4) | 0.36421 (12) | 0.0798 (13) |
| H5 | 0.396579 | 0.404315 | 0.371609 | 0.096* |
| C6 | 0.2621 (6) | 0.2855 (5) | 0.39534 (14) | 0.0977 (16) |
| H6 | 0.280074 | 0.303258 | 0.423272 | 0.117* |
| C7 | 0.1695 (6) | 0.2017 (4) | 0.38588 (17) | 0.0970 (16) |
| H7 | 0.124816 | 0.159862 | 0.407115 | 0.116* |
| C8 | 0.1405 (6) | 0.1773 (4) | 0.34539 (19) | 0.1091 (17) |
| H8 | 0.074177 | 0.119804 | 0.338752 | 0.131* |
| C9 | 0.2096 (5) | 0.2380 (4) | 0.31360 (14) | 0.0921 (14) |
| H9 | 0.188961 | 0.220341 | 0.285810 | 0.111* |
| C10 | 0.5138 (4) | 0.3654 (3) | 0.19787 (10) | 0.0543 (9) |
| C11 | 0.4240 (4) | 0.2731 (3) | 0.18940 (11) | 0.0677 (11) |
| H11 | 0.414767 | 0.214442 | 0.209129 | 0.081* |
| C12 | 0.3474 (4) | 0.2657 (4) | 0.15224 (13) | 0.0770 (12) |
| H12 | 0.285473 | 0.203753 | 0.147597 | 0.092* |
| C13 | 0.3629 (5) | 0.3501 (4) | 0.12213 (14) | 0.0828 (13) |
| H13 | 0.309935 | 0.346367 | 0.097415 | 0.099* |
| C14 | 0.4574 (5) | 0.4401 (4) | 0.12889 (13) | 0.0870 (14) |
| H14 | 0.471721 | 0.495095 | 0.108055 | 0.104* |
| C15 | 0.5317 (5) | 0.4495 (4) | 0.16667 (12) | 0.0744 (12) |
| H15 | 0.593370 | 0.511637 | 0.171223 | 0.089* |
| C16 | 0.7499 (4) | 0.3123 (3) | 0.22963 (9) | 0.0522 (9) |
| C17 | 0.8731 (5) | 0.3723 (4) | 0.21882 (12) | 0.0772 (12) |
| H17 | 0.870032 | 0.452549 | 0.217305 | 0.093* |
| C18 | 1.0013 (5) | 0.3154 (5) | 0.21019 (14) | 0.0910 (14) |
| H18 | 1.082786 | 0.357674 | 0.202763 | 0.109* |
| C19 | 1.0091 (5) | 0.1980 (5) | 0.21249 (13) | 0.0831 (13) |

| | | | | |
|---|---|---|---|---|
| H19 | 1.095450 | 0.159806 | 0.206974 | 0.100* |
| C20 | 0.8883 (5) | 0.1375 (4) | 0.22298 (12) | 0.0778 (12) |
| H20 | 0.891793 | 0.057265 | 0.224532 | 0.093* |
| C21 | 0.7618 (4) | 0.1940 (4) | 0.23126 (11) | 0.0703 (11) |
| H21 | 0.680645 | 0.150724 | 0.238279 | 0.084* |
| C22 | 0.6586 (4) | 0.2695 (3) | 0.33965 (10) | 0.0550 (9) |
| H22 | 0.620090 | 0.195694 | 0.336849 | 0.066* |
| C23 | 0.7494 (4) | 0.2969 (3) | 0.37590 (10) | 0.0496 (9) |
| C24 | 0.8122 (4) | 0.4061 (3) | 0.38002 (10) | 0.0526 (9) |
| C25 | 0.8931 (4) | 0.4348 (3) | 0.41580 (10) | 0.0500 (9) |
| C26 | 0.9079 (4) | 0.3519 (3) | 0.44648 (10) | 0.0529 (9) |
| H26 | 0.959861 | 0.370856 | 0.470443 | 0.064* |
| C27 | 0.8493 (4) | 0.2410 (3) | 0.44368 (10) | 0.0478 (8) |
| C28 | 0.7703 (4) | 0.2159 (3) | 0.40773 (10) | 0.0541 (9) |
| H28 | 0.730155 | 0.142665 | 0.404804 | 0.065* |
| C29 | 0.8755 (4) | 0.1531 (3) | 0.47686 (10) | 0.0504 (9) |
| C30 | 1.0031 (4) | 0.1530 (3) | 0.49976 (11) | 0.0604 (10) |
| H30 | 1.071466 | 0.210327 | 0.494851 | 0.072* |
| C31 | 1.0305 (4) | 0.0696 (4) | 0.52972 (11) | 0.0683 (11) |
| H31 | 1.116596 | 0.072379 | 0.544695 | 0.082* |
| C32 | 0.9326 (4) | −0.0180 (3) | 0.53792 (11) | 0.0622 (10) |
| C33 | 0.8045 (4) | −0.0174 (3) | 0.51594 (11) | 0.0621 (10) |
| H33 | 0.735757 | −0.074099 | 0.521325 | 0.075* |
| C34 | 0.7762 (4) | 0.0665 (3) | 0.48582 (10) | 0.0591 (10) |
| H34 | 0.688924 | 0.064645 | 0.471381 | 0.071* |
| C35 | 0.9668 (5) | −0.1114 (4) | 0.56979 (13) | 0.0922 (15) |
| H35A | 0.905661 | −0.102056 | 0.593855 | 0.138* |
| H35B | 1.066098 | −0.105268 | 0.578152 | 0.138* |
| H35C | 0.950142 | −0.185964 | 0.557520 | 0.138* |
| C36 | 0.9554 (4) | 0.5526 (3) | 0.42036 (11) | 0.0561 (10) |
| C37 | 1.0498 (4) | 0.5958 (3) | 0.39045 (11) | 0.0650 (10) |
| H37 | 1.074983 | 0.550160 | 0.367610 | 0.078* |
| C38 | 1.1070 (5) | 0.7055 (4) | 0.39409 (13) | 0.0770 (12) |
| H38 | 1.169606 | 0.732566 | 0.373533 | 0.092* |
| C39 | 1.0733 (6) | 0.7756 (4) | 0.42744 (16) | 0.0842 (14) |
| C40 | 0.9821 (6) | 0.7310 (4) | 0.45696 (15) | 0.0924 (14) |
| H40 | 0.958615 | 0.776139 | 0.480080 | 0.111* |
| C41 | 0.9239 (4) | 0.6224 (4) | 0.45388 (12) | 0.0730 (12) |
| H41 | 0.862259 | 0.595760 | 0.474761 | 0.088* |
| C42 | 1.1356 (8) | 0.8968 (4) | 0.43117 (18) | 0.143 (2) |
| H42A | 1.082749 | 0.948409 | 0.413288 | 0.214* |
| H42B | 1.235355 | 0.895979 | 0.422919 | 0.214* |
| H42C | 1.128080 | 0.922631 | 0.459675 | 0.214* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.090 (2) | 0.0512 (15) | 0.0548 (15) | −0.0053 (15) | −0.0084 (14) | −0.0028 (12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O2 | 0.092 (2) | 0.0583 (15) | 0.0569 (15) | −0.0119 (14) | −0.0203 (14) | 0.0123 (13) |
| N1 | 0.0618 (18) | 0.0565 (18) | 0.0413 (16) | 0.0019 (16) | −0.0026 (15) | 0.0023 (15) |
| C1 | 0.066 (2) | 0.045 (2) | 0.044 (2) | −0.001 (2) | −0.0056 (19) | −0.0016 (16) |
| C2 | 0.056 (2) | 0.048 (2) | 0.046 (2) | 0.0028 (19) | −0.0033 (18) | 0.0010 (16) |
| C3 | 0.071 (3) | 0.068 (2) | 0.053 (2) | 0.012 (2) | −0.007 (2) | 0.0017 (19) |
| C4 | 0.055 (2) | 0.059 (2) | 0.053 (2) | 0.010 (2) | −0.0002 (19) | −0.0080 (19) |
| C5 | 0.082 (3) | 0.093 (3) | 0.064 (3) | −0.016 (3) | 0.008 (2) | −0.016 (2) |
| C6 | 0.104 (4) | 0.123 (4) | 0.066 (3) | −0.014 (4) | 0.024 (3) | −0.006 (3) |
| C7 | 0.104 (4) | 0.081 (3) | 0.106 (4) | 0.005 (3) | 0.049 (3) | −0.008 (3) |
| C8 | 0.109 (4) | 0.098 (4) | 0.121 (4) | −0.033 (3) | 0.041 (4) | −0.039 (4) |
| C9 | 0.088 (3) | 0.111 (4) | 0.077 (3) | −0.018 (3) | 0.012 (3) | −0.034 (3) |
| C10 | 0.062 (2) | 0.053 (2) | 0.048 (2) | 0.0072 (19) | −0.0011 (18) | −0.0027 (18) |
| C11 | 0.078 (3) | 0.075 (3) | 0.050 (2) | −0.013 (2) | 0.000 (2) | 0.001 (2) |
| C12 | 0.077 (3) | 0.097 (3) | 0.057 (2) | −0.011 (3) | 0.001 (2) | −0.020 (3) |
| C13 | 0.086 (3) | 0.094 (3) | 0.068 (3) | 0.017 (3) | −0.022 (3) | −0.018 (3) |
| C14 | 0.112 (4) | 0.085 (3) | 0.064 (3) | 0.019 (3) | −0.020 (3) | 0.013 (2) |
| C15 | 0.090 (3) | 0.068 (3) | 0.065 (2) | 0.000 (2) | −0.017 (2) | 0.008 (2) |
| C16 | 0.055 (2) | 0.061 (2) | 0.0408 (19) | −0.007 (2) | −0.0051 (18) | −0.0024 (17) |
| C17 | 0.070 (3) | 0.083 (3) | 0.079 (3) | −0.007 (3) | 0.003 (2) | 0.008 (2) |
| C18 | 0.059 (3) | 0.115 (5) | 0.099 (4) | −0.016 (3) | 0.008 (3) | 0.001 (3) |
| C19 | 0.069 (3) | 0.109 (4) | 0.071 (3) | 0.018 (3) | −0.001 (2) | −0.009 (3) |
| C20 | 0.079 (3) | 0.075 (3) | 0.080 (3) | 0.010 (3) | 0.003 (3) | −0.007 (2) |
| C21 | 0.062 (3) | 0.066 (3) | 0.083 (3) | 0.000 (2) | 0.009 (2) | −0.001 (2) |
| C22 | 0.062 (2) | 0.054 (2) | 0.050 (2) | 0.0008 (19) | −0.0030 (19) | 0.0009 (18) |
| C23 | 0.056 (2) | 0.050 (2) | 0.0423 (19) | −0.0015 (18) | −0.0014 (17) | 0.0007 (17) |
| C24 | 0.061 (2) | 0.053 (2) | 0.044 (2) | 0.0029 (19) | 0.0006 (18) | 0.0043 (17) |
| C25 | 0.055 (2) | 0.053 (2) | 0.0423 (19) | 0.0028 (19) | 0.0005 (18) | −0.0002 (18) |
| C26 | 0.055 (2) | 0.061 (2) | 0.0427 (19) | 0.003 (2) | −0.0013 (17) | −0.0024 (18) |
| C27 | 0.050 (2) | 0.051 (2) | 0.0432 (19) | 0.0011 (18) | −0.0006 (17) | 0.0037 (16) |
| C28 | 0.058 (2) | 0.053 (2) | 0.052 (2) | −0.0032 (19) | −0.0009 (19) | 0.0029 (18) |
| C29 | 0.053 (2) | 0.054 (2) | 0.0452 (18) | 0.002 (2) | −0.0029 (19) | 0.0027 (17) |
| C30 | 0.058 (2) | 0.068 (3) | 0.055 (2) | −0.005 (2) | −0.0074 (19) | 0.009 (2) |
| C31 | 0.067 (3) | 0.080 (3) | 0.058 (2) | 0.002 (2) | −0.016 (2) | 0.008 (2) |
| C32 | 0.071 (3) | 0.062 (2) | 0.054 (2) | 0.008 (2) | −0.003 (2) | 0.0097 (19) |
| C33 | 0.063 (2) | 0.062 (3) | 0.062 (2) | −0.003 (2) | 0.000 (2) | 0.014 (2) |
| C34 | 0.058 (2) | 0.062 (2) | 0.058 (2) | −0.001 (2) | −0.0077 (19) | 0.007 (2) |
| C35 | 0.108 (4) | 0.088 (3) | 0.080 (3) | 0.011 (3) | −0.016 (3) | 0.032 (2) |
| C36 | 0.069 (3) | 0.051 (2) | 0.048 (2) | 0.001 (2) | −0.005 (2) | 0.0026 (18) |
| C37 | 0.090 (3) | 0.057 (2) | 0.049 (2) | −0.009 (2) | −0.005 (2) | −0.0032 (19) |
| C38 | 0.099 (3) | 0.068 (3) | 0.064 (3) | −0.015 (3) | −0.009 (2) | 0.010 (2) |
| C39 | 0.112 (4) | 0.052 (3) | 0.089 (3) | −0.014 (3) | −0.015 (3) | 0.002 (3) |
| C40 | 0.126 (4) | 0.062 (3) | 0.089 (3) | −0.001 (3) | 0.002 (3) | −0.026 (3) |
| C41 | 0.088 (3) | 0.063 (3) | 0.068 (3) | 0.000 (2) | 0.009 (2) | −0.013 (2) |
| C42 | 0.214 (7) | 0.064 (3) | 0.149 (5) | −0.046 (4) | −0.014 (5) | −0.014 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—H1 | 0.8200 | C20—H20 | 0.9300 |
| O1—C1 | 1.435 (4) | C20—C21 | 1.367 (5) |
| O2—H2 | 0.8200 | C21—H21 | 0.9300 |
| O2—C24 | 1.354 (4) | C22—H22 | 0.9300 |
| N1—C2 | 1.459 (4) | C22—C23 | 1.465 (4) |
| N1—C22 | 1.273 (4) | C23—C24 | 1.397 (4) |
| C1—C2 | 1.556 (4) | C23—C28 | 1.396 (4) |
| C1—C10 | 1.532 (5) | C24—C25 | 1.406 (4) |
| C1—C16 | 1.533 (5) | C25—C26 | 1.378 (4) |
| C2—H2A | 0.9800 | C25—C36 | 1.487 (5) |
| C2—C3 | 1.538 (5) | C26—H26 | 0.9300 |
| C3—H3A | 0.9700 | C26—C27 | 1.396 (4) |
| C3—H3B | 0.9700 | C27—C28 | 1.391 (4) |
| C3—C4 | 1.501 (5) | C27—C29 | 1.488 (4) |
| C4—C5 | 1.373 (5) | C28—H28 | 0.9300 |
| C4—C9 | 1.377 (5) | C29—C30 | 1.388 (4) |
| C5—H5 | 0.9300 | C29—C34 | 1.390 (4) |
| C5—C6 | 1.378 (5) | C30—H30 | 0.9300 |
| C6—H6 | 0.9300 | C30—C31 | 1.382 (5) |
| C6—C7 | 1.329 (6) | C31—H31 | 0.9300 |
| C7—H7 | 0.9300 | C31—C32 | 1.385 (5) |
| C7—C8 | 1.350 (6) | C32—C33 | 1.378 (5) |
| C8—H8 | 0.9300 | C32—C35 | 1.518 (5) |
| C8—C9 | 1.389 (6) | C33—H33 | 0.9300 |
| C9—H9 | 0.9300 | C33—C34 | 1.392 (5) |
| C10—C11 | 1.379 (5) | C34—H34 | 0.9300 |
| C10—C15 | 1.402 (5) | C35—H35A | 0.9600 |
| C11—H11 | 0.9300 | C35—H35B | 0.9600 |
| C11—C12 | 1.385 (5) | C35—H35C | 0.9600 |
| C12—H12 | 0.9300 | C36—C37 | 1.388 (5) |
| C12—C13 | 1.378 (5) | C36—C41 | 1.372 (5) |
| C13—H13 | 0.9300 | C37—H37 | 0.9300 |
| C13—C14 | 1.377 (6) | C37—C38 | 1.380 (5) |
| C14—H14 | 0.9300 | C38—H38 | 0.9300 |
| C14—C15 | 1.392 (5) | C38—C39 | 1.374 (6) |
| C15—H15 | 0.9300 | C39—C40 | 1.366 (6) |
| C16—C17 | 1.380 (5) | C39—C42 | 1.521 (6) |
| C16—C21 | 1.374 (5) | C40—H40 | 0.9300 |
| C17—H17 | 0.9300 | C40—C41 | 1.371 (5) |
| C17—C18 | 1.385 (6) | C41—H41 | 0.9300 |
| C18—H18 | 0.9300 | C42—H42A | 0.9600 |
| C18—C19 | 1.363 (6) | C42—H42B | 0.9600 |
| C19—H19 | 0.9300 | C42—H42C | 0.9600 |
| C19—C20 | 1.361 (6) | | |
| | | | |
| C1—O1—H1 | 109.5 | C16—C21—H21 | 118.7 |

| | | | |
|---|---|---|---|
| C24—O2—H2 | 109.5 | C20—C21—C16 | 122.6 (4) |
| C22—N1—C2 | 122.3 (3) | C20—C21—H21 | 118.7 |
| O1—C1—C2 | 107.4 (3) | N1—C22—H22 | 119.6 |
| O1—C1—C10 | 106.2 (3) | N1—C22—C23 | 120.7 (3) |
| O1—C1—C16 | 110.7 (3) | C23—C22—H22 | 119.6 |
| C10—C1—C2 | 113.9 (3) | C24—C23—C22 | 120.6 (3) |
| C10—C1—C16 | 108.4 (3) | C28—C23—C22 | 120.6 (3) |
| C16—C1—C2 | 110.2 (3) | C28—C23—C24 | 118.8 (3) |
| N1—C2—C1 | 106.1 (3) | O2—C24—C23 | 121.0 (3) |
| N1—C2—H2A | 109.8 | O2—C24—C25 | 118.2 (3) |
| N1—C2—C3 | 107.8 (3) | C23—C24—C25 | 120.8 (3) |
| C1—C2—H2A | 109.8 | C24—C25—C36 | 120.2 (3) |
| C3—C2—C1 | 113.5 (3) | C26—C25—C24 | 117.8 (3) |
| C3—C2—H2A | 109.8 | C26—C25—C36 | 122.0 (3) |
| C2—C3—H3A | 109.1 | C25—C26—H26 | 118.1 |
| C2—C3—H3B | 109.1 | C25—C26—C27 | 123.8 (3) |
| H3A—C3—H3B | 107.9 | C27—C26—H26 | 118.1 |
| C4—C3—C2 | 112.3 (3) | C26—C27—C29 | 121.3 (3) |
| C4—C3—H3A | 109.1 | C28—C27—C26 | 116.7 (3) |
| C4—C3—H3B | 109.1 | C28—C27—C29 | 122.0 (3) |
| C5—C4—C3 | 122.6 (4) | C23—C28—H28 | 118.9 |
| C5—C4—C9 | 116.4 (4) | C27—C28—C23 | 122.2 (3) |
| C9—C4—C3 | 121.0 (3) | C27—C28—H28 | 118.9 |
| C4—C5—H5 | 119.1 | C30—C29—C27 | 120.9 (3) |
| C4—C5—C6 | 121.7 (4) | C30—C29—C34 | 117.0 (3) |
| C6—C5—H5 | 119.1 | C34—C29—C27 | 122.1 (3) |
| C5—C6—H6 | 119.6 | C29—C30—H30 | 119.3 |
| C7—C6—C5 | 120.7 (5) | C31—C30—C29 | 121.4 (4) |
| C7—C6—H6 | 119.6 | C31—C30—H30 | 119.3 |
| C6—C7—H7 | 120.1 | C30—C31—H31 | 119.3 |
| C6—C7—C8 | 119.9 (5) | C30—C31—C32 | 121.5 (3) |
| C8—C7—H7 | 120.1 | C32—C31—H31 | 119.3 |
| C7—C8—H8 | 119.9 | C31—C32—C35 | 120.8 (4) |
| C7—C8—C9 | 120.2 (5) | C33—C32—C31 | 117.6 (3) |
| C9—C8—H8 | 119.9 | C33—C32—C35 | 121.6 (4) |
| C4—C9—C8 | 121.1 (4) | C32—C33—H33 | 119.4 |
| C4—C9—H9 | 119.4 | C32—C33—C34 | 121.2 (4) |
| C8—C9—H9 | 119.4 | C34—C33—H33 | 119.4 |
| C11—C10—C1 | 123.4 (3) | C29—C34—C33 | 121.4 (3) |
| C11—C10—C15 | 118.0 (3) | C29—C34—H34 | 119.3 |
| C15—C10—C1 | 118.3 (3) | C33—C34—H34 | 119.3 |
| C10—C11—H11 | 119.2 | C32—C35—H35A | 109.5 |
| C10—C11—C12 | 121.6 (4) | C32—C35—H35B | 109.5 |
| C12—C11—H11 | 119.2 | C32—C35—H35C | 109.5 |
| C11—C12—H12 | 120.0 | H35A—C35—H35B | 109.5 |
| C13—C12—C11 | 120.0 (4) | H35A—C35—H35C | 109.5 |
| C13—C12—H12 | 120.0 | H35B—C35—H35C | 109.5 |
| C12—C13—H13 | 120.2 | C37—C36—C25 | 120.5 (3) |

| | | | |
|---|---|---|---|
| C14—C13—C12 | 119.5 (4) | C41—C36—C25 | 122.3 (3) |
| C14—C13—H13 | 120.2 | C41—C36—C37 | 117.3 (4) |
| C13—C14—H14 | 119.7 | C36—C37—H37 | 119.5 |
| C13—C14—C15 | 120.5 (4) | C38—C37—C36 | 121.0 (4) |
| C15—C14—H14 | 119.7 | C38—C37—H37 | 119.5 |
| C10—C15—H15 | 119.9 | C37—C38—H38 | 119.3 |
| C14—C15—C10 | 120.2 (4) | C39—C38—C37 | 121.4 (4) |
| C14—C15—H15 | 119.9 | C39—C38—H38 | 119.3 |
| C17—C16—C1 | 121.5 (3) | C38—C39—C42 | 121.3 (5) |
| C21—C16—C1 | 122.1 (3) | C40—C39—C38 | 116.8 (4) |
| C21—C16—C17 | 116.4 (4) | C40—C39—C42 | 121.9 (5) |
| C16—C17—H17 | 119.4 | C39—C40—H40 | 118.7 |
| C16—C17—C18 | 121.2 (4) | C39—C40—C41 | 122.7 (4) |
| C18—C17—H17 | 119.4 | C41—C40—H40 | 118.7 |
| C17—C18—H18 | 119.7 | C36—C41—H41 | 119.6 |
| C19—C18—C17 | 120.6 (4) | C40—C41—C36 | 120.8 (4) |
| C19—C18—H18 | 119.7 | C40—C41—H41 | 119.6 |
| C18—C19—H19 | 120.6 | C39—C42—H42A | 109.5 |
| C20—C19—C18 | 118.8 (5) | C39—C42—H42B | 109.5 |
| C20—C19—H19 | 120.6 | C39—C42—H42C | 109.5 |
| C19—C20—H20 | 119.8 | H42A—C42—H42B | 109.5 |
| C19—C20—C21 | 120.3 (4) | H42A—C42—H42C | 109.5 |
| C21—C20—H20 | 119.8 | H42B—C42—H42C | 109.5 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2···N1 | 0.82 | 1.80 | 2.539 (4) | 148 |
| O1—H1···N1 | 0.82 | 2.46 | 2.763 (3) | 103 |