IUCrData

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# 4-Fluoro-2-({[(2*R*)-1-hydroxy-1,1,3-triphenylpropan-2-yl]imino}methyl)phenol

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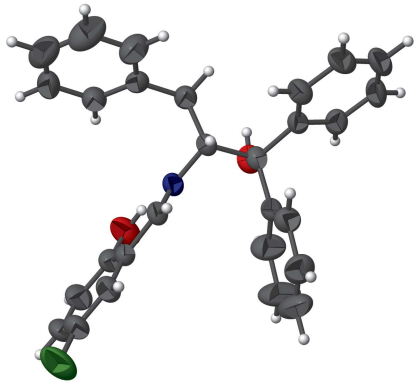
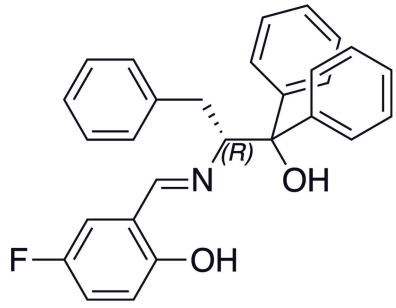
**Structural data**: full structural data are available from iucrdata.iucr.org

The title compound, $C_{28}H_{24}FNO_2$, crystallizes in the orthorhombic space group $P2_12_12_1$. A hydrogen-bonding network between the tertiary alcohol group and the fluoro substituent results in [010] chains in the solid state.

**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*)- or (*R*)-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). We routinely prepare both enantiomers of the ligands, and a number of them were examined by single-crystal X-ray diffraction, including the L-enantiomer of the title compound, in order to compare the structures of the free and bound ligand.

2-Hydroxy-5-fluoro-benzaldehyde 2*S*-(1,1,3-triphenylpropanol) imine, $C_{28}H_{24}FNO_2$, crystallizes in the orthorhombic space group $P2_12_12_1$ as shown in Fig. 1. The major structural features of the two enantiomers are similar, as expected. The L-enantiomer structure was collected at 100 K while the D-enantiomer was collected at 293 K. The unit-cell parameters in the current room-temperature structure are slightly larger (average 1.3%), presumably due to the higher temperature of the data collection. The absolute structure parameter of −0.1 (3) has a large uncertainty but the absolute configuration was verified by synthesis and polarimetry.

The compound has the expected imine–phenol structure as opposed to the iminium–phenoxide tautomer seen in derivatives with less steric bulk. The C23–C28 phenol aromatic ring is close to co-planar with atoms O2 [deviation from the ring plane = 0.040 (2) Å], C22 [–0.061 (2) Å], N1 [–0.034 (2) Å] and C2 [–0.039 (2) Å]. These four atoms exhibit less deviation from the plane than the enantiomer. The C22—N1—C2—C1 torsion angle is 110.2 (2)°, which places atom O1 1.555 (2) Å above the plane of the ring.

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2···N1 | 0.82 | 1.86 | 2.583 (3) | 147 |
| O1—H1···F1$^{i}$ | 0.82 | 2.94 | 3.720 (3) | 160 |
| C9—H9···F1$^{ii}$ | 0.93 | 2.54 | 3.467 (3) | 175 |
| C14—H14···O2$^{iii}$ | 0.93 | 2.58 | 3.369 (3) | 142 |

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$.

This deviation is 0.166 Å larger than that for the enantiomer at 100 K, although the torsion angle is almost identical.

The bonds between C27—C28, C23—C28 and C23—C24 are long at 1.39–1.41 Å while those between C24—C25, C25—C26 and C26—C27 are shorter at 1.36–1.37 Å. In contrast, the aromatic rings on the benzyl and phenyl substituents have typical C—C bond distances ranging from 1.37–1.39 Å. The aromatic C28—O2 bond at 1.349 (3) Å is substantially shorter than the aliphatic C1—O1 bond [1.439 (3) Å]. This bonding motif has been seen in related structures (Sha *et al.*, 2019).

There is an intramolecular O2—H2···N1 hydrogen bond (Table 1) between the salicylaldehyde alcohol group and the imine nitrogen atom, which closes an $S(6)$ ring and a long-range intermolecular hydrogen bond between the tertiary alcohol O1—H1 and the F1 atom of an adjacent molecule as shown in Fig. 2: the H···F and O···F distances are 2.94 and 3.720 (3) Å, respectively. Weak intermolecular C—H···F and C—H···O contacts are also observed.

## Synthesis and crystallization

Preparative details of the material have been reported previously (Sha *et al.*, 2019). Crystals in the form of light-yellow blocks were obtained by slow evaporation from the mixed solvents of hexane/ethyl acetate.

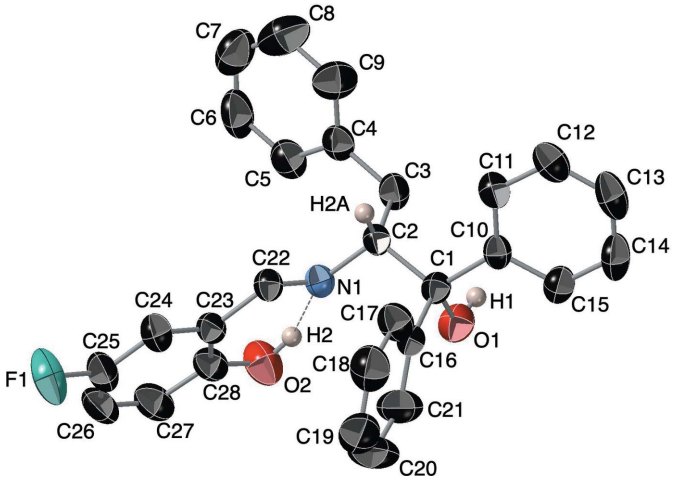



**Figure 1**
The asymmetric unit of the title compound with displacement ellipsoids shown at the 50% probability level. Hydrogen atoms besides H1, H2 and H2*A* have been omitted for clarity.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{28}H_{24}FNO_2$ |
| $M_r$ | 425.48 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ |
| Temperature (K) | 293 |
| $a, b, c$ (Å) | 6.0147 (2), 18.8172 (4), 20.4530 (5) |
| $V$ (Å$^3$) | 2314.87 (11) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.08 |
| Crystal size (mm) | 0.29 × 0.27 × 0.23 |
| | |
| Data collection | |
| Diffractometer | Rigaku XtaLAB Mini II |
| Absorption correction | Analytical [*CrysAlis PRO* (Rigaku OD, 2019); *ABSPACK* (Rigaku OD, 2017)] |
| $T_{min}$, $T_{max}$ | 0.995, 0.996 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 73250, 5721, 4529 |
| $R_{int}$ | 0.044 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.667 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.045, 0.098, 1.03 |
| No. of reflections | 5721 |
| No. of parameters | 291 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.14, −0.15 |
| Absolute structure | Flack $x$ determined using 1550 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) |
| Absolute structure parameter | −0.1 (3) |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2019), *SHELXS* (Sheldrick, 2008), *SHELXL* (Sheldrick, 2015), *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 wish to thank Eric W. Reinheimer for technical support.

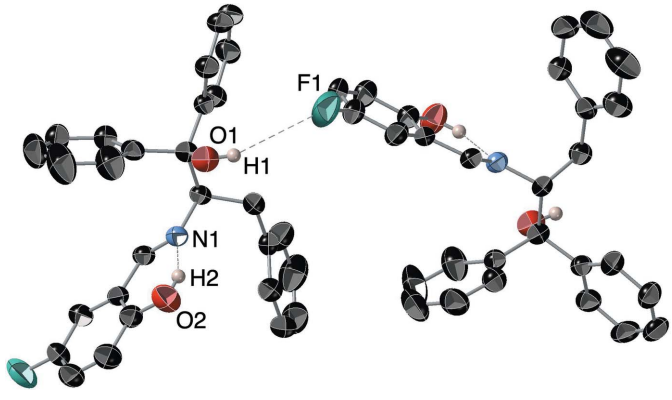



**Figure 2**
A view of the inter- and intramolecular hydrogen-bonding network along the *b* axis.

## Funding information

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# full crystallographic data

*IUCrData* (2020). **5**, x201580 [https://doi.org/10.1107/S2414314620015801]

## 4-Fluoro-2-({[(2*R*)-1-hydroxy-1,1,3-triphenylpropan-2-yl]imino}methyl)phenol

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### 4-Fluoro-2-({[(2*R*)-1-hydroxy-1,1,3-triphenylpropan-2-yl]imino}methyl)phenol

*Crystal data*

$C_{28}H_{24}FNO_2$
$M_r$ = 425.48
Orthorhombic, $P2_12_12_1$
$a$ = 6.0147 (2) Å
$b$ = 18.8172 (4) Å
$c$ = 20.4530 (5) Å
$V$ = 2314.87 (11) Å$^3$
$Z$ = 4
$F(000)$ = 896
$D_x$ = 1.221 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 20629 reflections
$\theta$ = 2.0–22.8°
$\mu$ = 0.08 mm$^{-1}$
$T$ = 293 K
Block, clear light yellow
0.29 × 0.27 × 0.23 mm

*Data collection*

Rigaku 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); *ABSPACK* (Rigaku OD, 2017)]
$T_{min}$ = 0.995, $T_{max}$ = 0.996
73250 measured reflections
5721 independent reflections
4529 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.044
$\theta_{max}$ = 28.3°, $\theta_{min}$ = 2.0°
$h$ = −8→8
$k$ = −25→25
$l$ = −27→27

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.098
$S$ = 1.03
5721 reflections
291 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2 + 0.2813P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.15 e Å$^{-3}$
Absolute structure: Flack $x$ determined using 1550 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.1 (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.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| F1 | 0.9178 (3) | 0.82714 (9) | 0.12710 (9) | 0.0962 (6) |
| O1 | 0.2298 (2) | 0.51725 (9) | 0.34826 (8) | 0.0552 (4) |
| H1 | 0.203611 | 0.474846 | 0.343181 | 0.083* |
| O2 | 0.2834 (3) | 0.62611 (10) | 0.17459 (9) | 0.0684 (5) |
| H2 | 0.328661 | 0.596034 | 0.200364 | 0.103* |
| N1 | 0.5550 (3) | 0.56695 (9) | 0.25587 (8) | 0.0413 (4) |
| C1 | 0.4599 (3) | 0.52649 (11) | 0.36563 (10) | 0.0398 (4) |
| C2 | 0.6059 (3) | 0.51204 (10) | 0.30374 (9) | 0.0386 (4) |
| H2A | 0.763405 | 0.515018 | 0.315703 | 0.046* |
| C3 | 0.5589 (4) | 0.43882 (11) | 0.27290 (10) | 0.0505 (5) |
| H3A | 0.581351 | 0.402322 | 0.305740 | 0.061* |
| H3B | 0.404359 | 0.436968 | 0.259445 | 0.061* |
| C4 | 0.7040 (4) | 0.42272 (10) | 0.21473 (10) | 0.0488 (5) |
| C5 | 0.6403 (5) | 0.44232 (13) | 0.15197 (12) | 0.0639 (7) |
| H5 | 0.505460 | 0.465438 | 0.145449 | 0.077* |
| C6 | 0.7771 (7) | 0.42755 (14) | 0.09884 (13) | 0.0835 (10) |
| H6 | 0.733148 | 0.441041 | 0.057053 | 0.100* |
| C7 | 0.9761 (7) | 0.39330 (17) | 0.10751 (18) | 0.0886 (11) |
| H7 | 1.067032 | 0.383652 | 0.071824 | 0.106* |
| C8 | 1.0393 (6) | 0.37362 (18) | 0.1685 (2) | 0.0909 (10) |
| H8 | 1.173814 | 0.350153 | 0.174553 | 0.109* |
| C9 | 0.9049 (5) | 0.38825 (15) | 0.22170 (14) | 0.0712 (7) |
| H9 | 0.951195 | 0.374498 | 0.263194 | 0.085* |
| C10 | 0.5143 (3) | 0.47796 (10) | 0.42366 (9) | 0.0402 (4) |
| C11 | 0.7181 (4) | 0.44477 (12) | 0.43239 (11) | 0.0495 (5) |
| H11 | 0.827905 | 0.449448 | 0.400675 | 0.059* |
| C12 | 0.7598 (5) | 0.40449 (12) | 0.48822 (11) | 0.0594 (6) |
| H12 | 0.896877 | 0.382316 | 0.493428 | 0.071* |
| C13 | 0.5999 (5) | 0.39739 (13) | 0.53549 (12) | 0.0657 (7) |
| H13 | 0.628493 | 0.370747 | 0.572854 | 0.079* |
| C14 | 0.3965 (5) | 0.42993 (14) | 0.52737 (11) | 0.0652 (7) |
| H14 | 0.287022 | 0.424937 | 0.559155 | 0.078* |
| C15 | 0.3550 (4) | 0.46988 (12) | 0.47220 (11) | 0.0520 (6) |
| H15 | 0.217525 | 0.491873 | 0.467401 | 0.062* |
| C16 | 0.4910 (4) | 0.60386 (11) | 0.38657 (10) | 0.0450 (5) |
| C17 | 0.6909 (5) | 0.62610 (12) | 0.41351 (12) | 0.0576 (6) |
| H17 | 0.803348 | 0.593111 | 0.420758 | 0.069* |
| C18 | 0.7254 (6) | 0.69653 (14) | 0.42975 (14) | 0.0764 (8) |
| H18 | 0.860574 | 0.710554 | 0.447662 | 0.092* |

| | | | | |
|---|---|---|---|---|
| C19 | 0.5618 (7) | 0.74562 (15) | 0.41960 (19) | 0.0930 (11) |
| H19 | 0.584347 | 0.792885 | 0.431100 | 0.112* |
| C20 | 0.3641 (7) | 0.72472 (17) | 0.3923 (2) | 0.1065 (13) |
| H20 | 0.253617 | 0.758201 | 0.384393 | 0.128* |
| C21 | 0.3279 (5) | 0.65428 (15) | 0.37652 (17) | 0.0797 (9) |
| H21 | 0.191920 | 0.640668 | 0.358886 | 0.096* |
| C22 | 0.6980 (4) | 0.61408 (10) | 0.24233 (9) | 0.0405 (4) |
| H22 | 0.837032 | 0.612055 | 0.262127 | 0.049* |
| C23 | 0.6501 (4) | 0.67165 (10) | 0.19634 (10) | 0.0415 (5) |
| C24 | 0.8092 (4) | 0.72373 (12) | 0.18420 (11) | 0.0534 (6) |
| H24 | 0.944724 | 0.723285 | 0.206112 | 0.064* |
| C25 | 0.7621 (5) | 0.77581 (12) | 0.13918 (11) | 0.0600 (6) |
| C26 | 0.5670 (5) | 0.77790 (13) | 0.10519 (12) | 0.0649 (7) |
| H26 | 0.542130 | 0.813151 | 0.074170 | 0.078* |
| C27 | 0.4081 (5) | 0.72738 (13) | 0.11734 (12) | 0.0643 (7) |
| H27 | 0.274328 | 0.728610 | 0.094574 | 0.077* |
| C28 | 0.4451 (4) | 0.67427 (11) | 0.16341 (11) | 0.0494 (5) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| F1 | 0.1196 (15) | 0.0738 (10) | 0.0954 (12) | −0.0342 (10) | −0.0053 (12) | 0.0365 (9) |
| O1 | 0.0371 (8) | 0.0709 (10) | 0.0577 (9) | −0.0053 (8) | −0.0042 (7) | 0.0022 (8) |
| O2 | 0.0602 (11) | 0.0715 (11) | 0.0735 (12) | −0.0102 (9) | −0.0239 (9) | 0.0191 (9) |
| N1 | 0.0450 (10) | 0.0434 (9) | 0.0355 (8) | −0.0008 (8) | −0.0026 (7) | 0.0031 (7) |
| C1 | 0.0344 (10) | 0.0472 (11) | 0.0379 (10) | −0.0011 (9) | −0.0006 (9) | 0.0030 (8) |
| C2 | 0.0407 (11) | 0.0398 (10) | 0.0354 (9) | −0.0013 (8) | 0.0003 (9) | 0.0055 (8) |
| C3 | 0.0633 (15) | 0.0433 (11) | 0.0447 (11) | −0.0084 (11) | 0.0065 (11) | 0.0011 (9) |
| C4 | 0.0647 (15) | 0.0364 (10) | 0.0451 (12) | −0.0073 (10) | 0.0022 (11) | −0.0033 (8) |
| C5 | 0.0920 (19) | 0.0495 (12) | 0.0503 (14) | 0.0007 (14) | 0.0024 (14) | 0.0016 (11) |
| C6 | 0.144 (3) | 0.0599 (15) | 0.0463 (14) | −0.013 (2) | 0.0211 (18) | 0.0026 (11) |
| C7 | 0.108 (3) | 0.0727 (19) | 0.085 (2) | −0.018 (2) | 0.041 (2) | −0.0203 (17) |
| C8 | 0.073 (2) | 0.090 (2) | 0.109 (3) | 0.0033 (18) | 0.013 (2) | −0.035 (2) |
| C9 | 0.0765 (19) | 0.0720 (16) | 0.0652 (16) | 0.0083 (15) | −0.0036 (15) | −0.0167 (13) |
| C10 | 0.0460 (12) | 0.0388 (10) | 0.0358 (10) | −0.0093 (9) | −0.0003 (9) | 0.0010 (8) |
| C11 | 0.0504 (13) | 0.0506 (11) | 0.0476 (12) | −0.0049 (11) | −0.0025 (10) | 0.0081 (9) |
| C12 | 0.0697 (17) | 0.0526 (13) | 0.0557 (14) | −0.0031 (12) | −0.0173 (14) | 0.0105 (11) |
| C13 | 0.099 (2) | 0.0578 (14) | 0.0406 (12) | −0.0197 (15) | −0.0153 (14) | 0.0125 (11) |
| C14 | 0.089 (2) | 0.0683 (15) | 0.0383 (12) | −0.0223 (15) | 0.0110 (13) | 0.0044 (11) |
| C15 | 0.0564 (14) | 0.0538 (12) | 0.0458 (12) | −0.0077 (11) | 0.0086 (11) | −0.0009 (10) |
| C16 | 0.0505 (13) | 0.0439 (11) | 0.0408 (10) | 0.0046 (10) | 0.0119 (10) | 0.0044 (9) |
| C17 | 0.0706 (17) | 0.0487 (12) | 0.0537 (13) | −0.0050 (12) | −0.0038 (12) | −0.0010 (10) |
| C18 | 0.098 (2) | 0.0607 (15) | 0.0702 (17) | −0.0204 (17) | 0.0091 (17) | −0.0113 (13) |
| C19 | 0.122 (3) | 0.0468 (15) | 0.110 (3) | −0.0052 (19) | 0.050 (2) | −0.0141 (16) |
| C20 | 0.103 (3) | 0.0553 (17) | 0.162 (4) | 0.0271 (19) | 0.033 (3) | 0.001 (2) |
| C21 | 0.0665 (19) | 0.0636 (16) | 0.109 (2) | 0.0174 (15) | 0.0101 (17) | −0.0004 (16) |
| C22 | 0.0433 (11) | 0.0440 (10) | 0.0342 (9) | −0.0007 (9) | −0.0013 (9) | 0.0033 (8) |
| C23 | 0.0537 (13) | 0.0385 (10) | 0.0323 (9) | 0.0028 (9) | 0.0016 (9) | 0.0003 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C24 | 0.0629 (15) | 0.0514 (12) | 0.0458 (12) | −0.0055 (11) | −0.0026 (11) | 0.0075 (10) |
| C25 | 0.0844 (18) | 0.0432 (12) | 0.0522 (13) | −0.0071 (12) | 0.0050 (14) | 0.0087 (10) |
| C26 | 0.096 (2) | 0.0462 (12) | 0.0527 (14) | 0.0142 (14) | −0.0048 (15) | 0.0117 (10) |
| C27 | 0.0745 (18) | 0.0602 (14) | 0.0581 (14) | 0.0113 (14) | −0.0164 (14) | 0.0101 (12) |
| C28 | 0.0578 (14) | 0.0465 (11) | 0.0439 (11) | 0.0045 (11) | −0.0071 (11) | 0.0011 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F1—C25 | 1.368 (3) | C12—H12 | 0.9300 |
| O1—H1 | 0.8200 | C12—C13 | 1.370 (4) |
| O1—C1 | 1.439 (3) | C13—H13 | 0.9300 |
| O2—H2 | 0.8200 | C13—C14 | 1.378 (4) |
| O2—C28 | 1.349 (3) | C14—H14 | 0.9300 |
| N1—C2 | 1.456 (2) | C14—C15 | 1.379 (3) |
| N1—C22 | 1.266 (3) | C15—H15 | 0.9300 |
| C1—C2 | 1.564 (3) | C16—C17 | 1.387 (3) |
| C1—C10 | 1.533 (3) | C16—C21 | 1.380 (3) |
| C1—C16 | 1.529 (3) | C17—H17 | 0.9300 |
| C2—H2A | 0.9800 | C17—C18 | 1.382 (3) |
| C2—C3 | 1.541 (3) | C18—H18 | 0.9300 |
| C3—H3A | 0.9700 | C18—C19 | 1.365 (5) |
| C3—H3B | 0.9700 | C19—H19 | 0.9300 |
| C3—C4 | 1.506 (3) | C19—C20 | 1.371 (5) |
| C4—C5 | 1.389 (3) | C20—H20 | 0.9300 |
| C4—C9 | 1.379 (4) | C20—C21 | 1.382 (5) |
| C5—H5 | 0.9300 | C21—H21 | 0.9300 |
| C5—C6 | 1.391 (4) | C22—H22 | 0.9300 |
| C6—H6 | 0.9300 | C22—C23 | 1.463 (3) |
| C6—C7 | 1.371 (5) | C23—C24 | 1.392 (3) |
| C7—H7 | 0.9300 | C23—C28 | 1.406 (3) |
| C7—C8 | 1.355 (5) | C24—H24 | 0.9300 |
| C8—H8 | 0.9300 | C24—C25 | 1.374 (3) |
| C8—C9 | 1.384 (4) | C25—C26 | 1.364 (4) |
| C9—H9 | 0.9300 | C26—H26 | 0.9300 |
| C10—C11 | 1.387 (3) | C26—C27 | 1.371 (4) |
| C10—C15 | 1.388 (3) | C27—H27 | 0.9300 |
| C11—H11 | 0.9300 | C27—C28 | 1.391 (3) |
| C11—C12 | 1.393 (3) | | |

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|---|---|---|---|
| C1—O1—H1 | 109.5 | C12—C13—H13 | 120.2 |
| C28—O2—H2 | 109.5 | C12—C13—C14 | 119.7 (2) |
| C22—N1—C2 | 120.07 (17) | C14—C13—H13 | 120.2 |
| O1—C1—C2 | 108.61 (16) | C13—C14—H14 | 120.0 |
| O1—C1—C10 | 108.93 (16) | C13—C14—C15 | 120.1 (2) |
| O1—C1—C16 | 107.55 (17) | C15—C14—H14 | 120.0 |
| C10—C1—C2 | 113.77 (16) | C10—C15—H15 | 119.3 |
| C16—C1—C2 | 108.88 (15) | C14—C15—C10 | 121.4 (2) |
| C16—C1—C10 | 108.92 (16) | C14—C15—H15 | 119.3 |

| | | | |
|---|---|---|---|
| N1—C2—C1 | 107.61 (15) | C17—C16—C1 | 120.29 (19) |
| N1—C2—H2A | 109.3 | C21—C16—C1 | 121.7 (2) |
| N1—C2—C3 | 108.70 (16) | C21—C16—C17 | 117.9 (2) |
| C1—C2—H2A | 109.3 | C16—C17—H17 | 119.5 |
| C3—C2—C1 | 112.55 (16) | C18—C17—C16 | 121.0 (3) |
| C3—C2—H2A | 109.3 | C18—C17—H17 | 119.5 |
| C2—C3—H3A | 108.9 | C17—C18—H18 | 119.9 |
| C2—C3—H3B | 108.9 | C19—C18—C17 | 120.3 (3) |
| H3A—C3—H3B | 107.7 | C19—C18—H18 | 119.9 |
| C4—C3—C2 | 113.37 (18) | C18—C19—H19 | 120.2 |
| C4—C3—H3A | 108.9 | C18—C19—C20 | 119.5 (3) |
| C4—C3—H3B | 108.9 | C20—C19—H19 | 120.2 |
| C5—C4—C3 | 121.1 (2) | C19—C20—H20 | 119.8 |
| C9—C4—C3 | 121.4 (2) | C19—C20—C21 | 120.5 (3) |
| C9—C4—C5 | 117.5 (2) | C21—C20—H20 | 119.8 |
| C4—C5—H5 | 119.8 | C16—C21—C20 | 120.9 (3) |
| C4—C5—C6 | 120.4 (3) | C16—C21—H21 | 119.6 |
| C6—C5—H5 | 119.8 | C20—C21—H21 | 119.6 |
| C5—C6—H6 | 119.7 | N1—C22—H22 | 119.2 |
| C7—C6—C5 | 120.6 (3) | N1—C22—C23 | 121.70 (19) |
| C7—C6—H6 | 119.7 | C23—C22—H22 | 119.2 |
| C6—C7—H7 | 120.2 | C24—C23—C22 | 120.0 (2) |
| C8—C7—C6 | 119.5 (3) | C24—C23—C28 | 119.53 (19) |
| C8—C7—H7 | 120.2 | C28—C23—C22 | 120.43 (19) |
| C7—C8—H8 | 119.8 | C23—C24—H24 | 120.7 |
| C7—C8—C9 | 120.4 (3) | C25—C24—C23 | 118.6 (2) |
| C9—C8—H8 | 119.8 | C25—C24—H24 | 120.7 |
| C4—C9—C8 | 121.6 (3) | F1—C25—C24 | 118.9 (3) |
| C4—C9—H9 | 119.2 | C26—C25—F1 | 118.5 (2) |
| C8—C9—H9 | 119.2 | C26—C25—C24 | 122.6 (2) |
| C11—C10—C1 | 123.82 (18) | C25—C26—H26 | 120.4 |
| C11—C10—C15 | 117.93 (19) | C25—C26—C27 | 119.2 (2) |
| C15—C10—C1 | 118.15 (19) | C27—C26—H26 | 120.4 |
| C10—C11—H11 | 119.7 | C26—C27—H27 | 119.7 |
| C10—C11—C12 | 120.6 (2) | C26—C27—C28 | 120.6 (2) |
| C12—C11—H11 | 119.7 | C28—C27—H27 | 119.7 |
| C11—C12—H12 | 119.8 | O2—C28—C23 | 121.85 (19) |
| C13—C12—C11 | 120.3 (3) | O2—C28—C27 | 118.8 (2) |
| C13—C12—H12 | 119.8 | C27—C28—C23 | 119.3 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2···N1 | 0.82 | 1.86 | 2.583 (3) | 147 |
| O1—H1···F1$^{i}$ | 0.82 | 2.94 | 3.720 (3) | 160 |

| | | | | |
|---|---|---|---|---|
| C9—H9···F1$^{ii}$ | 0.93 | 2.54 | 3.467 (3) | 175 |
| C14—H14···O2$^{iii}$ | 0.93 | 2.58 | 3.369 (3) | 142 |

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) −*x*+2, *y*−1/2, −*z*+1/2; (iii) −*x*+1/2, −*y*+1, *z*+1/2.