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# (1*R*,3*S*)-Methyl 2-benzyl-6,7-dimethoxy-1-phenyl-1,2,3,4-tetrahydroisoquinoline-3-carboxylate

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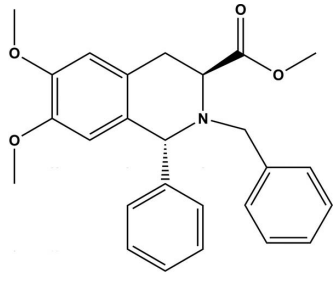
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.028; $wR$ factor = 0.077; data-to-parameter ratio = 12.7.

In the title compound, $C_{26}H_{27}NO_4$, a precursor to novel chiral catalysts, the N-containing six-membered ring assumes a half-boat conformation. Various C—H$\cdots\pi$ interactions and intermolecular short contacts (C$\cdots$H = 2.81–2.90 Å) link the molecules together in the crystal structure.

## Related literature

For the synthesis, see: Chakka *et al.* (2009). For crystallograhic details of analogous molecules, see Alberch *et al.* (2004); Aubry *et al.* (2006).



## Experimental

### Crystal data

$C_{26}H_{27}NO_4$
$M_r$ = 417.49
Triclinic, $P\overline{1}$
$a$ = 6.0199 (1) Å
$b$ = 9.2592 (2) Å
$c$ = 11.0429 (2) Å
$\alpha$ = 73.365 (1)°
$\beta$ = 74.694 (1)°
$\gamma$ = 75.737 (1)°
$V$ = 559.05 (2) Å$^3$
$Z$ = 1
Cu $K\alpha$ radiation
$\mu$ = 0.67 mm$^{-1}$
$T$ = 173 K
0.22 × 0.12 × 0.08 mm

### Data collection

Bruker Kappa Duo APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min}$ = 0.692, $T_{max}$ = 0.753
7546 measured reflections
3561 independent reflections
3536 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.012

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.077
$S$ = 1.07
3561 reflections
281 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$
Absolute structure: Flack (1983), 1483 Friedel pairs
Flack parameter: −0.01 (14)

**Table 1**
C—H$\cdots\pi$ interaction (Å, °).

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| C19—H19$A\cdots Cg^{i}$ | 0.98 | 2.82 | 3.639 (2) | 148 |

Symmetry code: (i) $x+1, y+1, z-1$. $Cg$ is the centroid of the C21–C26 ring.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) and *X-SEED* (Barbour, 2001); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *ORTEP-3*.

The authors wish to thank Dr Hong Su of the Chemistry Department at the University of Cape Town for his assistance with the crystallographic data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2607).

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# supporting information

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## (1*R*,3*S*)-Methyl 2-benzyl-6,7-dimethoxy-1-phenyl-1,2,3,4-tetrahydroisoquinoline-3-carboxylate

**Tricia Naicker, Michael McKay, Thavendran Govender, Hendrik G. Kruger and Glenn E. M. Maguire**

### S1. Comment

The title compound (2, Scheme 1) is a precursor in the synthesis of novel chiral ligands containing a tetrahydroisoquinoline backbone. We have recently reported the use of these ligands as successful catalysts for transfer hydrogenations reactions (Chakka *et al.*, 2009).

Compound 2 was derived from commercially available *L*-DOPA and benzaldehyde. Diastereomers formed during the first step of the synthesis were separated to yield subsequent derivatives and the title compound and with the stereochemistry as illustrated in Figure 1. The absolute stereochemistry was confirmed to be *R,S* at C1 and C9 positions respectively.

From the crystal structure it is evident that the N-containing six membered ring assumes a half boat conformation (Figure 1). This differs from an analogous structure which assumes a half chair conformation (Aubry *et al.*, 2006 and Alberch *et al.*, 2004). A possible reason for this change in conformation could be either the introduction of a substitutent on the nitrogen or the *trans* position of the phenyl ring at the C1 position.

The molecule exhibits various intermolecular short contacts *i.e*. between the methyl ester hydrogen (H11C) and phenyl ring (C14) of a neighbouring molecule; H15 to C6 and C7 and H24 to C14 and C15.

The methoxy groups display different interactions. The first methoxy group at C4 position displays one interaction between H18B and O2, which is the ether oxygen of the other methoxy group. The second methoxy group at C5 position displays three interactions; the first being the above mentioned interaction with H18B and O2, the second being a short contact between O2 and H25, and the third being another CH-$\pi$ the interaction between H19A and C25. The atoms involved in these short contacts are shown in Figure 2.

### S2. Experimental

To a solution of **1** (Scheme 1) (500 mg, 1.52 mmol) in acetonitrile (20 ml), solid $K_2CO_3$ (635 mg, 4.58 mmol) was added followed by benzyl bromide (286 mg, 1.67 mmol) at ambient temperature. There after the reaction mixture was refluxed for 3 h. Completion of the reaction was monitored with TLC using hexane/ethyl acetate (60:40, $R_f$ 1/2). The solvent was evaporated and 30 ml of ethylacetate was added, washed with 2 × 10 ml of water, the organic layer was separated, and dried over anhydrous $MgSO_4$. The solvent was evaporated under reduced pressure to afford crude product, which was purified by column chromatography using hexane/ethyl acetate (60:40) as the eluent to yield pure benzyl ester **2** (0.44 g, 90%) as a white solid.

$^1$H NMR (400 MHz/$CDCl_3$): $\delta$ = 7.44 (d, *J* = 1.16 Hz, 2H), 7.32–7.10 (m, 14H), 7.0–6.88 (m, 6H), 6.69 (s, 1H), 6.38 (s, 1H), 4.74 (s,1*H*), 4.21 (d, *J* = 13.60 Hz, 1H), 4.14 (q, *J* = 3.70, 12.74 Hz, 1H), 3.89 (s, 3H), 3.72 (s, 3H), 3.57 (s, 1H),

3.30–3.18 (m, 2H), 2.60 (dd, $J$ = 3.60, 16.48 Hz, 1H).

Colourless crystals suitable for X-ray diffraction were obtained by slow evaporation of **2** in dichloromethane at room temperature.

### S3. Refinement

Hydrogen atoms, first located in the difference map, were positioned geometrically and allowed to ride on their respective parent atoms, with C—H bond lengths of 1.00 (CH), 0.99 ($CH_2$), 0.98 ($CH_3$) or 0.93 (aromatic CH). They were then refined with a riding model with $U_{iso}$(H) = 1.5$U_{eq}$($CH_3$) and $U_{iso}$(H) = 1.2$U_{eq}$($X$) for $X$ = CH or $CH_2$. The largest residual electron density peak of 0.16 e/Å$^3$ is 0.86 Å from O4.

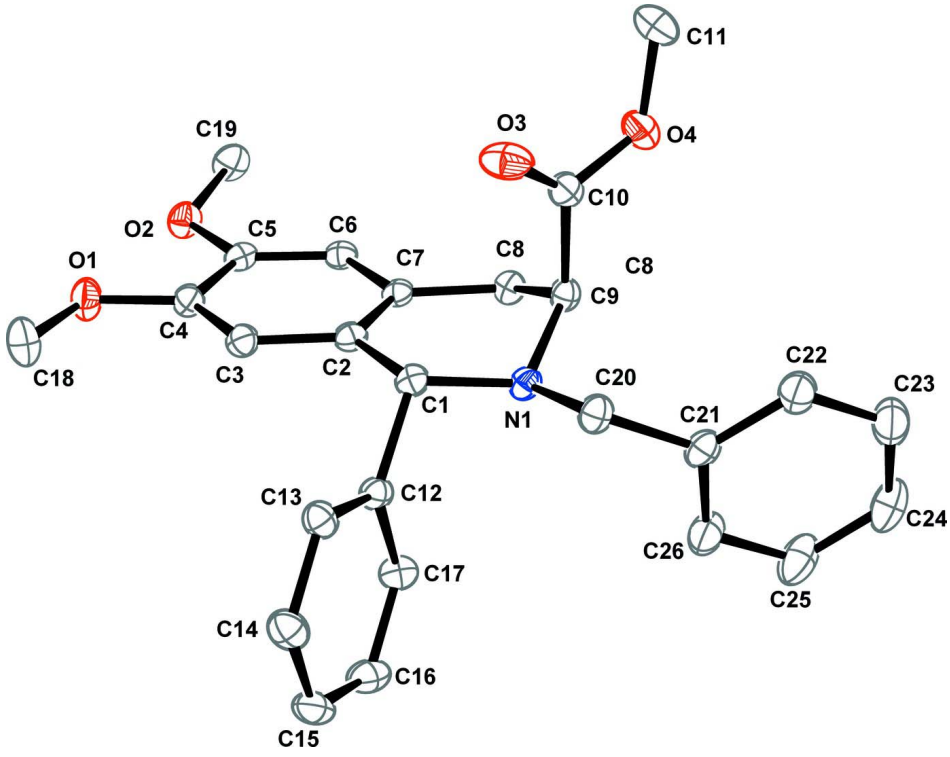


**Figure 1**

Molecular structure of the title compound **2** showing numbering scheme. Displacement elipsoids are drawn at the 30% probability level. Hydrogen atoms are omitted for clarity.

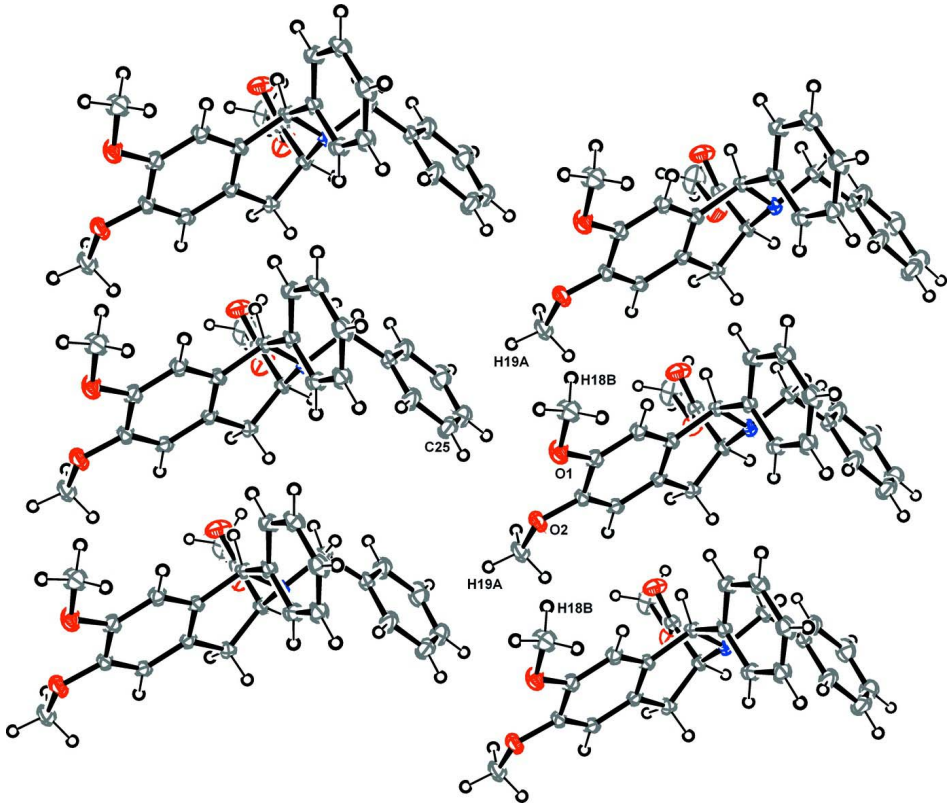


**Figure 2**

Intermolecular short contact interactions present in the crystal structure of the title compound **2**. The atoms involved in the interactions (as discussed) are labelled. different interactions displayed by methoxy groups. Displacement ellipsoids are drawn at the 30% probability level, and the hydrogen atoms appear as speheres of arbitrary radii.

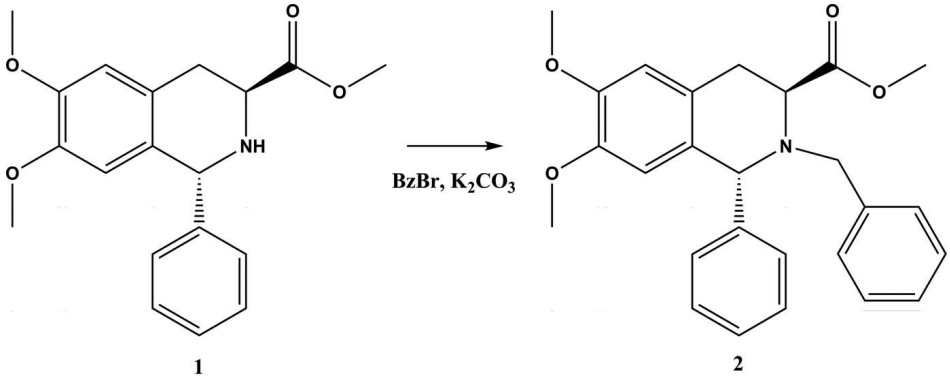


**Figure 3**

The formation of the title compound.

## (1*R*,3*S*)-Methyl 2-benzyl-6,7-dimethoxy-1-phenyl-1,2,3,4- tetrahydroisoquinoline-3-carboxylate

*Crystal data*

$C_{26}H_{27}NO_4$
$M_r = 417.49$
Triclinic, $P\overline{1}$
Hall symbol: P 1
$a = 6.0199$ (1) Å
$b = 9.2592$ (2) Å
$c = 11.0429$ (2) Å
$\alpha = 73.365$ (1)°
$\beta = 74.694$ (1)°
$\gamma = 75.737$ (1)°
$V = 559.05$ (2) Å$^3$
$Z = 1$
$F(000) = 222$
$D_x = 1.240$ Mg m$^{-3}$
Melting point: 420 K
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 7546 reflections
$\theta = 4.3$–$68.9°$
$\mu = 0.67$ mm$^{-1}$
$T = 173$ K
Needle, yellow
0.22 × 0.12 × 0.08 mm

*Data collection*

Bruker Kappa Duo APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
0.5° $\varphi$ scans and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min} = 0.692$, $T_{max} = 0.753$
7546 measured reflections
3561 independent reflections
3536 reflections with $I > 2\sigma(I)$
$R_{int} = 0.012$
$\theta_{max} = 68.9°$, $\theta_{min} = 4.3°$
$h = -6 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 13$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.077$
$S = 1.07$
3561 reflections
281 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.0785P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å$^{-3}$
$\Delta\rho_{min} = -0.16$ e Å$^{-3}$
Extinction correction: *SHELXS97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0426 (18)
Absolute structure: Flack (1983), 1483 Friedel pairs
Absolute structure parameter: −0.01 (14)

*Special details*

**Experimental**. Half sphere of data collected using *SAINT* strategy (Bruker, 2006). Crystal to detector distance = 50 mm; combination of $\varphi$ and $\omega$ scans of 0.5°, 80 s per °, 2 iterations.

**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.

**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 | 0.2537 (2) | 0.14310 (13) | 0.46249 (10) | 0.0396 (3) |
| O2 | 0.51646 (19) | 0.27489 (12) | 0.53134 (10) | 0.0364 (3) |
| O3 | −0.3602 (2) | 0.20138 (13) | 1.02179 (14) | 0.0468 (3) |
| O4 | −0.1914 (3) | 0.23948 (15) | 1.16480 (12) | 0.0514 (4) |
| N1 | −0.0675 (2) | −0.10502 (13) | 1.05031 (11) | 0.0262 (3) |
| C1 | −0.1027 (2) | −0.07812 (15) | 0.91791 (14) | 0.0247 (3) |
| H1 | −0.2641 | −0.0179 | 0.9133 | 0.030* |
| C2 | 0.0745 (2) | 0.01232 (15) | 0.81875 (14) | 0.0256 (3) |
| C3 | 0.0848 (3) | 0.03078 (16) | 0.68640 (14) | 0.0283 (3) |
| H3 | −0.0128 | −0.0169 | 0.6612 | 0.034* |
| C4 | 0.2340 (3) | 0.11668 (16) | 0.59283 (14) | 0.0298 (3) |
| C5 | 0.3790 (2) | 0.18793 (15) | 0.63010 (14) | 0.0280 (3) |
| C6 | 0.3717 (2) | 0.16708 (16) | 0.75934 (14) | 0.0270 (3) |
| H6 | 0.4725 | 0.2122 | 0.7846 | 0.032* |
| C7 | 0.2187 (2) | 0.08063 (15) | 0.85461 (13) | 0.0252 (3) |
| C8 | 0.2148 (2) | 0.06332 (17) | 0.99497 (14) | 0.0292 (3) |
| H8A | 0.2370 | 0.1606 | 1.0067 | 0.035* |
| H8B | 0.3468 | −0.0180 | 1.0206 | 0.035* |
| C9 | −0.0138 (3) | 0.02263 (16) | 1.08230 (14) | 0.0269 (3) |
| H9 | 0.0152 | −0.0155 | 1.1721 | 0.032* |
| C10 | −0.2107 (3) | 0.16235 (17) | 1.08378 (14) | 0.0308 (3) |
| C11 | −0.3714 (5) | 0.3725 (2) | 1.1814 (2) | 0.0633 (6) |
| H11A | −0.3393 | 0.4199 | 1.2422 | 0.095* |
| H11B | −0.5244 | 0.3412 | 1.2153 | 0.095* |
| H11C | −0.3725 | 0.4466 | 1.0979 | 0.095* |
| C12 | −0.0842 (3) | −0.23317 (16) | 0.89052 (13) | 0.0263 (3) |
| C13 | −0.2621 (3) | −0.26446 (17) | 0.84954 (14) | 0.0293 (3) |
| H13 | −0.3966 | −0.1876 | 0.8363 | 0.035* |
| C14 | −0.2445 (3) | −0.40782 (18) | 0.82777 (16) | 0.0346 (3) |
| H14 | −0.3673 | −0.4285 | 0.7998 | 0.042* |
| C15 | −0.0496 (3) | −0.52049 (17) | 0.84649 (16) | 0.0368 (4) |
| H15 | −0.0378 | −0.6184 | 0.8315 | 0.044* |
| C16 | 0.1286 (3) | −0.48936 (17) | 0.88734 (16) | 0.0382 (4) |
| H16 | 0.2632 | −0.5662 | 0.9002 | 0.046* |
| C17 | 0.1116 (3) | −0.34743 (17) | 0.90937 (16) | 0.0330 (3) |
| H17 | 0.2344 | −0.3274 | 0.9377 | 0.040* |
| C18 | 0.1052 (3) | 0.0782 (2) | 0.41938 (17) | 0.0462 (4) |
| H18A | 0.1349 | 0.1062 | 0.3247 | 0.069* |
| H18B | −0.0591 | 0.1176 | 0.4541 | 0.069* |
| H18C | 0.1379 | −0.0337 | 0.4497 | 0.069* |
| C19 | 0.6262 (3) | 0.3736 (2) | 0.56531 (18) | 0.0429 (4) |
| H19A | 0.7198 | 0.4294 | 0.4872 | 0.064* |
| H19B | 0.7283 | 0.3123 | 0.6249 | 0.064* |
| H19C | 0.5059 | 0.4469 | 0.6072 | 0.064* |
| C20 | −0.2513 (3) | −0.17390 (17) | 1.15037 (14) | 0.0304 (3) |

| | | | | |
|---|---|---|---|---|
| H20A | −0.2927 | −0.2542 | 1.1219 | 0.037* |
| H20B | −0.3934 | −0.0944 | 1.1635 | 0.037* |
| C21 | −0.1694 (3) | −0.24341 (17) | 1.27560 (14) | 0.0311 (3) |
| C22 | −0.2758 (3) | −0.1877 (2) | 1.38479 (16) | 0.0404 (4) |
| H22 | −0.4093 | −0.1075 | 1.3826 | 0.049* |
| C23 | −0.1904 (4) | −0.2472 (2) | 1.49690 (17) | 0.0506 (5) |
| H23 | −0.2661 | −0.2086 | 1.5714 | 0.061* |
| C24 | 0.0038 (4) | −0.3620 (2) | 1.50087 (18) | 0.0536 (5) |
| H24 | 0.0642 | −0.4016 | 1.5775 | 0.064* |
| C25 | 0.1101 (4) | −0.4193 (3) | 1.39405 (19) | 0.0553 (5) |
| H25 | 0.2436 | −0.4994 | 1.3971 | 0.066* |
| C26 | 0.0240 (3) | −0.3612 (2) | 1.28165 (17) | 0.0442 (4) |
| H26 | 0.0980 | −0.4022 | 1.2082 | 0.053* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0486 (7) | 0.0478 (7) | 0.0253 (5) | −0.0190 (5) | −0.0072 (5) | −0.0048 (5) |
| O2 | 0.0368 (6) | 0.0380 (6) | 0.0331 (6) | −0.0168 (5) | −0.0028 (5) | −0.0019 (4) |
| O3 | 0.0365 (6) | 0.0370 (6) | 0.0757 (9) | 0.0013 (5) | −0.0246 (6) | −0.0222 (6) |
| O4 | 0.0722 (10) | 0.0442 (7) | 0.0412 (6) | 0.0050 (6) | −0.0192 (6) | −0.0217 (5) |
| N1 | 0.0261 (6) | 0.0264 (6) | 0.0261 (6) | −0.0075 (5) | −0.0070 (5) | −0.0023 (5) |
| C1 | 0.0222 (7) | 0.0244 (7) | 0.0275 (7) | −0.0042 (5) | −0.0065 (6) | −0.0050 (5) |
| C2 | 0.0230 (7) | 0.0222 (6) | 0.0291 (7) | −0.0020 (5) | −0.0053 (6) | −0.0044 (5) |
| C3 | 0.0290 (7) | 0.0276 (7) | 0.0295 (7) | −0.0058 (6) | −0.0086 (6) | −0.0058 (6) |
| C4 | 0.0312 (8) | 0.0294 (8) | 0.0275 (7) | −0.0033 (6) | −0.0076 (6) | −0.0052 (6) |
| C5 | 0.0250 (7) | 0.0236 (7) | 0.0311 (7) | −0.0033 (5) | −0.0040 (6) | −0.0027 (5) |
| C6 | 0.0230 (7) | 0.0245 (7) | 0.0337 (8) | −0.0024 (5) | −0.0085 (6) | −0.0065 (6) |
| C7 | 0.0228 (7) | 0.0222 (7) | 0.0297 (7) | −0.0006 (5) | −0.0088 (6) | −0.0049 (6) |
| C8 | 0.0263 (8) | 0.0325 (7) | 0.0302 (7) | −0.0075 (6) | −0.0081 (6) | −0.0058 (6) |
| C9 | 0.0275 (7) | 0.0291 (7) | 0.0250 (6) | −0.0072 (5) | −0.0069 (6) | −0.0050 (5) |
| C10 | 0.0316 (8) | 0.0305 (7) | 0.0298 (7) | −0.0109 (6) | −0.0013 (6) | −0.0065 (6) |
| C11 | 0.0849 (16) | 0.0475 (11) | 0.0494 (11) | 0.0069 (10) | −0.0024 (11) | −0.0257 (9) |
| C12 | 0.0268 (7) | 0.0247 (7) | 0.0253 (7) | −0.0068 (5) | −0.0022 (6) | −0.0040 (5) |
| C13 | 0.0283 (8) | 0.0314 (7) | 0.0285 (7) | −0.0065 (6) | −0.0038 (6) | −0.0085 (6) |
| C14 | 0.0344 (8) | 0.0366 (8) | 0.0369 (8) | −0.0141 (6) | −0.0029 (7) | −0.0126 (6) |
| C15 | 0.0429 (9) | 0.0267 (7) | 0.0385 (8) | −0.0109 (7) | 0.0038 (7) | −0.0115 (6) |
| C16 | 0.0353 (8) | 0.0269 (8) | 0.0444 (9) | −0.0015 (6) | −0.0021 (7) | −0.0057 (6) |
| C17 | 0.0285 (7) | 0.0303 (8) | 0.0387 (8) | −0.0053 (6) | −0.0058 (6) | −0.0069 (6) |
| C18 | 0.0482 (10) | 0.0640 (12) | 0.0332 (8) | −0.0173 (8) | −0.0102 (8) | −0.0148 (8) |
| C19 | 0.0440 (9) | 0.0401 (9) | 0.0446 (9) | −0.0217 (7) | −0.0066 (8) | −0.0005 (7) |
| C20 | 0.0289 (7) | 0.0335 (7) | 0.0287 (7) | −0.0100 (6) | −0.0052 (6) | −0.0042 (6) |
| C21 | 0.0307 (8) | 0.0308 (8) | 0.0297 (7) | −0.0122 (6) | −0.0048 (6) | 0.0001 (6) |
| C22 | 0.0433 (9) | 0.0407 (9) | 0.0340 (8) | −0.0071 (7) | −0.0067 (7) | −0.0055 (7) |
| C23 | 0.0641 (13) | 0.0568 (11) | 0.0323 (8) | −0.0186 (9) | −0.0081 (8) | −0.0083 (8) |
| C24 | 0.0515 (11) | 0.0696 (13) | 0.0350 (9) | −0.0169 (10) | −0.0173 (8) | 0.0073 (8) |
| C25 | 0.0405 (10) | 0.0633 (12) | 0.0423 (10) | 0.0024 (8) | −0.0087 (8) | 0.0080 (9) |
| C26 | 0.0414 (10) | 0.0460 (10) | 0.0322 (8) | 0.0004 (7) | −0.0032 (7) | −0.0005 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C4 | 1.3668 (18) | C12—C17 | 1.393 (2) |
| O1—C18 | 1.428 (2) | C13—C14 | 1.390 (2) |
| O2—C5 | 1.3681 (17) | C13—H13 | 0.9500 |
| O2—C19 | 1.428 (2) | C14—C15 | 1.382 (2) |
| O3—C10 | 1.197 (2) | C14—H14 | 0.9500 |
| O4—C10 | 1.3363 (19) | C15—C16 | 1.385 (3) |
| O4—C11 | 1.446 (2) | C15—H15 | 0.9500 |
| N1—C9 | 1.4538 (18) | C16—C17 | 1.379 (2) |
| N1—C20 | 1.4667 (18) | C16—H16 | 0.9500 |
| N1—C1 | 1.4743 (18) | C17—H17 | 0.9500 |
| C1—C12 | 1.5222 (19) | C18—H18A | 0.9800 |
| C1—C2 | 1.5285 (19) | C18—H18B | 0.9800 |
| C1—H1 | 1.0000 | C18—H18C | 0.9800 |
| C2—C7 | 1.381 (2) | C19—H19A | 0.9800 |
| C2—C3 | 1.408 (2) | C19—H19B | 0.9800 |
| C3—C4 | 1.378 (2) | C19—H19C | 0.9800 |
| C3—H3 | 0.9500 | C20—C21 | 1.505 (2) |
| C4—C5 | 1.412 (2) | C20—H20A | 0.9900 |
| C5—C6 | 1.375 (2) | C20—H20B | 0.9900 |
| C6—C7 | 1.402 (2) | C21—C22 | 1.384 (2) |
| C6—H6 | 0.9500 | C21—C26 | 1.387 (2) |
| C7—C8 | 1.506 (2) | C22—C23 | 1.381 (3) |
| C8—C9 | 1.521 (2) | C22—H22 | 0.9500 |
| C8—H8A | 0.9900 | C23—C24 | 1.374 (3) |
| C8—H8B | 0.9900 | C23—H23 | 0.9500 |
| C9—C10 | 1.525 (2) | C24—C25 | 1.369 (3) |
| C9—H9 | 1.0000 | C24—H24 | 0.9500 |
| C11—H11A | 0.9800 | C25—C26 | 1.385 (3) |
| C11—H11B | 0.9800 | C25—H25 | 0.9500 |
| C11—H11C | 0.9800 | C26—H26 | 0.9500 |
| C12—C13 | 1.386 (2) | | |
| | | | |
| C4—O1—C18 | 117.43 (12) | C17—C12—C1 | 119.97 (13) |
| C5—O2—C19 | 116.91 (12) | C12—C13—C14 | 120.24 (14) |
| C10—O4—C11 | 116.59 (16) | C12—C13—H13 | 119.9 |
| C9—N1—C20 | 111.85 (11) | C14—C13—H13 | 119.9 |
| C9—N1—C1 | 116.21 (11) | C15—C14—C13 | 120.45 (15) |
| C20—N1—C1 | 113.54 (11) | C15—C14—H14 | 119.8 |
| N1—C1—C12 | 108.07 (11) | C13—C14—H14 | 119.8 |
| N1—C1—C2 | 111.52 (11) | C14—C15—C16 | 119.39 (14) |
| C12—C1—C2 | 111.33 (11) | C14—C15—H15 | 120.3 |
| N1—C1—H1 | 108.6 | C16—C15—H15 | 120.3 |
| C12—C1—H1 | 108.6 | C17—C16—C15 | 120.36 (14) |
| C2—C1—H1 | 108.6 | C17—C16—H16 | 119.8 |
| C7—C2—C3 | 119.04 (13) | C15—C16—H16 | 119.8 |
| C7—C2—C1 | 122.20 (12) | C16—C17—C12 | 120.60 (15) |

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| C3—C2—C1 | 118.73 (13) | C16—C17—H17 | 119.7 |
| C4—C3—C2 | 121.22 (14) | C12—C17—H17 | 119.7 |
| C4—C3—H3 | 119.4 | O1—C18—H18A | 109.5 |
| C2—C3—H3 | 119.4 | O1—C18—H18B | 109.5 |
| O1—C4—C3 | 125.46 (14) | H18A—C18—H18B | 109.5 |
| O1—C4—C5 | 115.04 (12) | O1—C18—H18C | 109.5 |
| C3—C4—C5 | 119.50 (13) | H18A—C18—H18C | 109.5 |
| O2—C5—C6 | 125.15 (14) | H18B—C18—H18C | 109.5 |
| O2—C5—C4 | 115.76 (13) | O2—C19—H19A | 109.5 |
| C6—C5—C4 | 119.09 (13) | O2—C19—H19B | 109.5 |
| C5—C6—C7 | 121.42 (14) | H19A—C19—H19B | 109.5 |
| C5—C6—H6 | 119.3 | O2—C19—H19C | 109.5 |
| C7—C6—H6 | 119.3 | H19A—C19—H19C | 109.5 |
| C2—C7—C6 | 119.71 (13) | H19B—C19—H19C | 109.5 |
| C2—C7—C8 | 120.93 (12) | N1—C20—C21 | 110.54 (12) |
| C6—C7—C8 | 119.36 (13) | N1—C20—H20A | 109.5 |
| C7—C8—C9 | 112.16 (12) | C21—C20—H20A | 109.5 |
| C7—C8—H8A | 109.2 | N1—C20—H20B | 109.5 |
| C9—C8—H8A | 109.2 | C21—C20—H20B | 109.5 |
| C7—C8—H8B | 109.2 | H20A—C20—H20B | 108.1 |
| C9—C8—H8B | 109.2 | C22—C21—C26 | 118.49 (15) |
| H8A—C8—H8B | 107.9 | C22—C21—C20 | 121.50 (15) |
| N1—C9—C8 | 109.50 (12) | C26—C21—C20 | 119.94 (15) |
| N1—C9—C10 | 115.01 (12) | C23—C22—C21 | 120.77 (17) |
| C8—C9—C10 | 112.26 (12) | C23—C22—H22 | 119.6 |
| N1—C9—H9 | 106.5 | C21—C22—H22 | 119.6 |
| C8—C9—H9 | 106.5 | C24—C23—C22 | 120.14 (18) |
| C10—C9—H9 | 106.5 | C24—C23—H23 | 119.9 |
| O3—C10—O4 | 123.72 (15) | C22—C23—H23 | 119.9 |
| O3—C10—C9 | 126.64 (14) | C25—C24—C23 | 119.84 (18) |
| O4—C10—C9 | 109.64 (13) | C25—C24—H24 | 120.1 |
| O4—C11—H11A | 109.5 | C23—C24—H24 | 120.1 |
| O4—C11—H11B | 109.5 | C24—C25—C26 | 120.33 (18) |
| H11A—C11—H11B | 109.5 | C24—C25—H25 | 119.8 |
| O4—C11—H11C | 109.5 | C26—C25—H25 | 119.8 |
| H11A—C11—H11C | 109.5 | C25—C26—C21 | 120.42 (17) |
| H11B—C11—H11C | 109.5 | C25—C26—H26 | 119.8 |
| C13—C12—C17 | 118.96 (13) | C21—C26—H26 | 119.8 |
| C13—C12—C1 | 121.05 (13) | | |

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|---|---|---|---|
| C9—N1—C1—C12 | 163.45 (12) | C1—N1—C9—C10 | 65.95 (16) |
| C20—N1—C1—C12 | −64.73 (15) | C7—C8—C9—N1 | 49.38 (16) |
| C9—N1—C1—C2 | 40.77 (16) | C7—C8—C9—C10 | −79.63 (15) |
| C20—N1—C1—C2 | 172.59 (11) | C11—O4—C10—O3 | 3.4 (2) |
| N1—C1—C2—C7 | −10.29 (17) | C11—O4—C10—C9 | −177.57 (15) |
| C12—C1—C2—C7 | −131.08 (14) | N1—C9—C10—O3 | −28.2 (2) |
| N1—C1—C2—C3 | 171.92 (11) | C8—C9—C10—O3 | 97.86 (18) |
| C12—C1—C2—C3 | 51.13 (17) | N1—C9—C10—O4 | 152.77 (13) |

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|---|---|---|---|
| C7—C2—C3—C4 | −0.7 (2) | C8—C9—C10—O4 | −81.16 (15) |
| C1—C2—C3—C4 | 177.19 (12) | N1—C1—C12—C13 | 125.53 (14) |
| C18—O1—C4—C3 | 0.9 (2) | C2—C1—C12—C13 | −111.68 (14) |
| C18—O1—C4—C5 | −177.87 (13) | N1—C1—C12—C17 | −53.04 (16) |
| C2—C3—C4—O1 | −178.91 (14) | C2—C1—C12—C17 | 69.75 (16) |
| C2—C3—C4—C5 | −0.1 (2) | C17—C12—C13—C14 | 0.0 (2) |
| C19—O2—C5—C6 | −12.4 (2) | C1—C12—C13—C14 | −178.56 (13) |
| C19—O2—C5—C4 | 167.07 (13) | C12—C13—C14—C15 | −0.1 (2) |
| O1—C4—C5—O2 | 0.83 (18) | C13—C14—C15—C16 | 0.1 (2) |
| C3—C4—C5—O2 | −178.07 (12) | C14—C15—C16—C17 | 0.1 (2) |
| O1—C4—C5—C6 | −179.67 (13) | C15—C16—C17—C12 | −0.2 (2) |
| C3—C4—C5—C6 | 1.4 (2) | C13—C12—C17—C16 | 0.2 (2) |
| O2—C5—C6—C7 | 177.51 (12) | C1—C12—C17—C16 | 178.75 (14) |
| C4—C5—C6—C7 | −1.9 (2) | C9—N1—C20—C21 | −64.04 (15) |
| C3—C2—C7—C6 | 0.19 (19) | C1—N1—C20—C21 | 162.04 (12) |
| C1—C2—C7—C6 | −177.59 (12) | N1—C20—C21—C22 | 115.75 (16) |
| C3—C2—C7—C8 | −179.65 (13) | N1—C20—C21—C26 | −61.09 (19) |
| C1—C2—C7—C8 | 2.57 (19) | C26—C21—C22—C23 | 0.5 (3) |
| C5—C6—C7—C2 | 1.1 (2) | C20—C21—C22—C23 | −176.41 (16) |
| C5—C6—C7—C8 | −179.02 (13) | C21—C22—C23—C24 | 0.7 (3) |
| C2—C7—C8—C9 | −22.20 (18) | C22—C23—C24—C25 | −1.2 (3) |
| C6—C7—C8—C9 | 157.96 (12) | C23—C24—C25—C26 | 0.6 (3) |
| C20—N1—C9—C8 | 165.87 (12) | C24—C25—C26—C21 | 0.6 (3) |
| C1—N1—C9—C8 | −61.53 (16) | C22—C21—C26—C25 | −1.1 (3) |
| C20—N1—C9—C10 | −66.65 (15) | C20—C21—C26—C25 | 175.85 (17) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C19—H19*A*···*Cg*$^{i}$ | 0.98 | 2.82 | 3.639 (2) | 148 |

Symmetry code: (i) *x*+1, *y*+1, *z*−1.