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# *rac*-(3*S*,4*S*)-3-Hydroxy-4-phenyl-1-[(*S*)-(3-phenyl-4,5-dihydro-1,2-oxazol-5-yl)-methyl]-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one

**Mohamed Rida,[a]* El Mokhtar Essassi,[a,b] Stéphane Massip,[c] Saïd Lazar[d] and Hafid Zouihri[e]**

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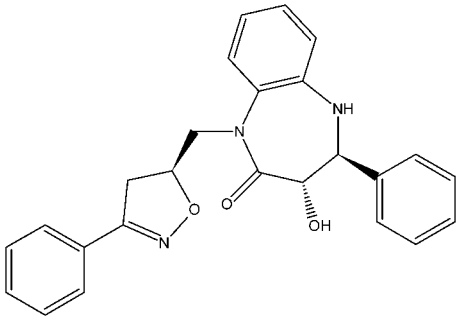
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.050; $wR$ factor = 0.157; data-to-parameter ratio = 12.5.

In the title compound, $C_{25}H_{23}N_3O_3$, the seven-membered diazepine ring adopts a boat conformation with the hydroxy-substituted C atom at the prow and fused-ring C atoms at the stern. The crystal packing features C—H···O, C—H···$\pi$ and N—H ···$\pi$ interactions

## Related literature

For the preparation and biological activity of benzodiazepines, see: Ahabchane *et al.* (1999), Grunewald *et al.* (1996); Ding *et al.*, (1999). For related structures, see: Saber *et al.* (2010*a*,*b*); Ballo *et al.* (2010). For puckering parameters, see: Cremer & Pople (1975).




## Experimental

### Crystal data

$C_{25}H_{23}N_3O_3$
$M_r$ = 413.46
Triclinic, $P\overline{1}$
$a$ = 8.981 (2) Å
$b$ = 9.044 (2) Å
$c$ = 13.685 (1) Å
$\alpha$ = 95.373 (10)°
$\beta$ = 102.433 (10)°
$\gamma$ = 100.03 (2)°
$V$ = 1058.9 (3) Å$^3$
$Z$ = 2
Cu $K\alpha$ radiation
$\mu$ = 0.70 mm$^{-1}$
$T$ = 293 K
0.15 × 0.10 × 0.08 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.903, $T_{max}$ = 0.946
3584 measured reflections
3584 independent reflections
3232 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.000
2 standard reflections every 90 min
intensity decay: none

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.157
$S$ = 1.05
3584 reflections
286 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

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

*Cg*4 is the centroid of the C26–C31 ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C16—H16···O22 | 0.93 | 2.44 | 3.206 (3) | 140 |
| C25—H25*a*···O14$^{i}$ | 0.97 | 2.59 | 3.493 (3) | 154 |
| C27—H27···O15$^{ii}$ | 0.93 | 2.40 | 3.302 (3) | 162 |
| C28—H28···O22$^{i}$ | 0.93 | 2.52 | 3.152 (2) | 125 |
| C19—H19···*Cg*4$^{iii}$ | 0.93 | 2.78 | 3.534 (3) | 139 |
| N13—H13···*Cg*4$^{iii}$ | 0.93 (2) | 2.77 (2) | 3.671 (3) | 164 |

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LD2003).

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# supporting information

*Acta Cryst.* (2011). E**67**, o945–o946 [doi:10.1107/S1600536811010129]

## *rac*-(3*S*,4*S*)-3-Hydroxy-4-phenyl-1-[(*S*)-(3-phenyl-4,5-dihydro-1,2-oxazol-5-yl)methyl]-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one

**Mohamed Rida, El Mokhtar Essassi, Stéphane Massip, Saïd Lazar and Hafid Zouihri**

### S1. Comment

Benzodiazipines have found wide-spread clinical use as inhibitors of phenylethanolamine N-methyltransferase [Grunewald *et al.*, 1996] and inhibitors of farnesyltransferase [Ding *et al.*, 1999]. In view of their importance and to ascertain molecular conformation [Ahabchane *et al.*, 1999. Saber *et al.* (2010*a* and 2010*b*) and Ballo *et al.* (2010)], a crystallographic study of the title compound has been carried out.

The molecular structure of the title compound is depicted in Fig.1. The seven-membred ring adopts a boat conformation with equatorial orientations of both phenyl and hydroxyl substituents. Its puckering parameters [Cremer & Pople, 1975] are $q_2$ = 0.997 (2) Å, $q_3$ = 0.1541 (19) Å, $\varphi_2$ = 317.05 (11)°, $\varphi_3$ = 185.2 (8)°.

Phenyl substituent at C24 atom is rotated out of the plane of the 4,5-dihydro-1,2-oxazole ring by 20.1 (1)°.

The crystal packing is controlled by C—H···O H-bonds and C—H ···$\pi$ and N—H ···$\pi$ interactions involving the C26-C31 (Cg4) benzene ring (Table 1).

### S2. Experimental

A solution of NaOCl (10 ml) was added to a mixture of 1-allylmethyl-3-hydroxy-4-phenyltetrahydro-1,5-benzodiazepin-2-one (2.58 g, 0.01 mmol) and benzaldoxime (1.21 g, 0.01 mmol) in chloroform (30 ml) under vigorous stirring. After 3 h, the organic layer was washed with water and dried by addition of anhydrous $MgSO_4$. After removal of the solvent by evaporation, the crude residual mixture obtained was purified by column chromatography on a silica gel column and eluted with an eluent composed of ether/petroleum ether (1/9). Crystals was obtained when solvent was allowed to evaporate.

### S3. Refinement

All O–H and C—H H-atoms were placed in calculated positions (C—H: 0.93 to 0.98 Å and O–H = 0.82) and allowed to ride on their parent atoms, with U (H) set to 1.2 U(C) and 1.5 U(O). Torsion angle for the hydroxyl group was optimized from electron density.

The amino H-atom was located in a difference Fourier map and was refined independently.

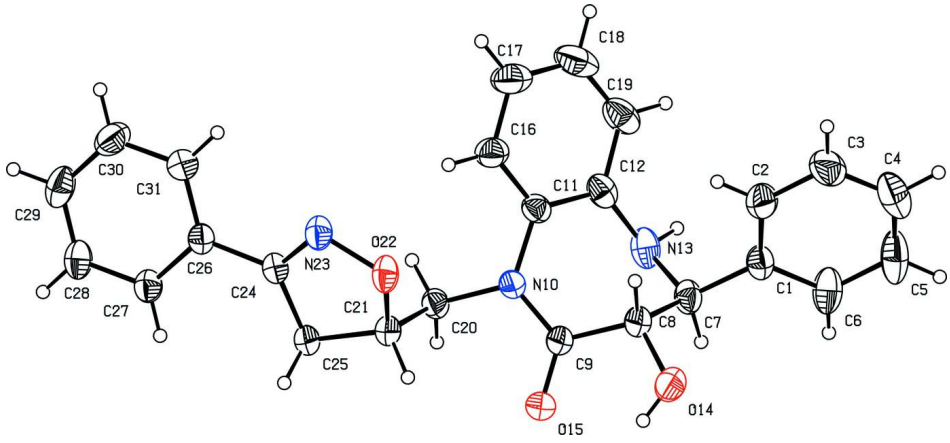


**Figure 1**

Molecular view of the title compound showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of an arbitrary radius.

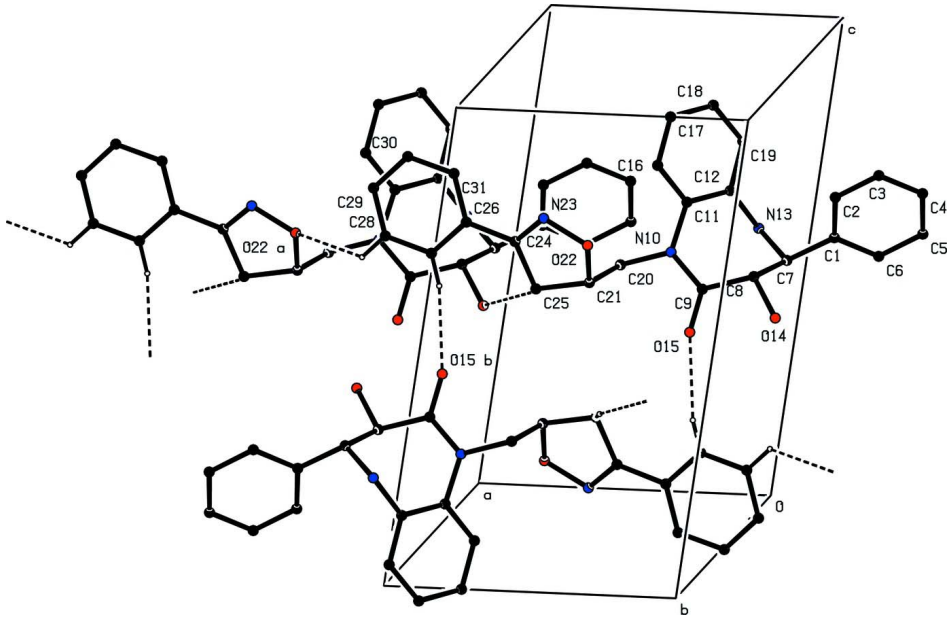


**Figure 2**

Partial packing view showing chains formed by C—H···O hydrogen bondings. H atoms not involved in the hydrogen bonds have been omitted for clarity.

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*Crystal data*

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Hall symbol: -P 1
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$c = 13.685$ (1) Å
$\alpha = 95.373$ (10)°
$\beta = 102.433$ (10)°
$\gamma = 100.03$ (2)°
$V = 1058.9$ (3) Å$^3$
$Z = 2$

$F(000) = 436$
$D_x = 1.297$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54180$ Å
Cell parameters from 25 reflections
$\theta = 25–35°$
$\mu = 0.70$ mm$^{-1}$
$T = 293$ K
Prism, colourless
0.15 × 0.10 × 0.08 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$–$2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min} = 0.903$, $T_{max} = 0.946$
3584 measured reflections
3584 independent reflections
3232 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 64.9°$, $\theta_{min} = 5.0°$
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = 0 \rightarrow 16$
2 standard reflections every 90 min
intensity decay: none

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.157$
$S = 1.05$
3584 reflections
286 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0902P)^2 + 0.363P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.20$ e Å$^{-3}$
$\Delta\rho_{min} = -0.17$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.086 (5)

*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.

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O14 | −0.14283 (18) | 0.8107 (2) | 0.54940 (13) | 0.0717 (5) |
| O15 | 0.1367 (2) | 0.8258 (3) | 0.51556 (12) | 0.0931 (7) |
| O22 | 0.45964 (14) | 1.08367 (16) | 0.74426 (11) | 0.0562 (4) |
| N10 | 0.25123 (17) | 0.78999 (18) | 0.67247 (11) | 0.0479 (4) |
| N13 | 0.0378 (2) | 0.52139 (19) | 0.67025 (15) | 0.0621 (5) |
| N23 | 0.61410 (18) | 1.11628 (19) | 0.80393 (13) | 0.0534 (4) |
| C1 | −0.2338 (2) | 0.5566 (2) | 0.66492 (17) | 0.0546 (5) |
| C2 | −0.2327 (3) | 0.6165 (3) | 0.76241 (19) | 0.0742 (7) |
| C3 | −0.3594 (3) | 0.5798 (4) | 0.8033 (2) | 0.0860 (8) |

| | | | | |
|---|---|---|---|---|
| C4 | −0.4916 (3) | 0.4842 (3) | 0.7485 (3) | 0.0888 (9) |
| C5 | −0.4981 (3) | 0.4269 (3) | 0.6516 (3) | 0.0944 (10) |
| C6 | −0.3691 (3) | 0.4630 (3) | 0.6095 (2) | 0.0768 (7) |
| C7 | −0.0924 (2) | 0.5884 (2) | 0.62070 (16) | 0.0550 (5) |
| C8 | −0.0343 (2) | 0.7579 (2) | 0.62179 (15) | 0.0521 (5) |
| C9 | 0.1256 (2) | 0.7912 (2) | 0.59864 (14) | 0.0560 (5) |
| C11 | 0.2384 (2) | 0.7330 (2) | 0.76507 (14) | 0.0478 (5) |
| C12 | 0.1330 (2) | 0.5989 (2) | 0.76299 (17) | 0.0543 (5) |
| C16 | 0.3373 (3) | 0.8063 (3) | 0.85519 (15) | 0.0599 (5) |
| C17 | 0.3345 (3) | 0.7461 (4) | 0.94383 (18) | 0.0787 (8) |
| C18 | 0.2311 (4) | 0.6129 (4) | 0.9423 (2) | 0.0884 (9) |
| C19 | 0.1311 (3) | 0.5418 (3) | 0.8540 (2) | 0.0733 (7) |
| C20 | 0.4057 (2) | 0.8317 (2) | 0.65084 (14) | 0.0497 (5) |
| C21 | 0.4573 (2) | 0.9998 (2) | 0.64844 (14) | 0.0492 (5) |
| C24 | 0.7062 (2) | 1.0913 (2) | 0.74752 (14) | 0.0455 (4) |
| C25 | 0.6252 (2) | 1.0381 (2) | 0.63910 (14) | 0.0505 (5) |
| C26 | 0.8751 (2) | 1.1172 (2) | 0.78925 (14) | 0.0460 (4) |
| C27 | 0.9750 (2) | 1.1281 (2) | 0.72391 (16) | 0.0529 (5) |
| C28 | 1.1338 (2) | 1.1536 (3) | 0.76154 (19) | 0.0634 (6) |
| C29 | 1.1953 (3) | 1.1658 (3) | 0.8629 (2) | 0.0721 (7) |
| C30 | 1.0978 (3) | 1.1550 (3) | 0.92885 (19) | 0.0741 (7) |
| C31 | 0.9385 (3) | 1.1308 (3) | 0.89236 (16) | 0.0611 (5) |
| H2 | −0.1443 | 0.6829 | 0.8010 | 0.089* |
| H3 | −0.3548 | 0.6208 | 0.8691 | 0.103* |
| H4 | −0.5762 | 0.4584 | 0.7768 | 0.107* |
| H5 | −0.5886 | 0.3635 | 0.6131 | 0.113* |
| H6 | −0.3752 | 0.4230 | 0.5433 | 0.092* |
| H7 | −0.1244 | 0.5420 | 0.5498 | 0.066* |
| H8 | −0.0283 | 0.8109 | 0.6888 | 0.062* |
| H13 | 0.001 (3) | 0.421 (2) | 0.677 (2) | 0.090 (8)* |
| H14 | −0.1016 | 0.8399 | 0.5048 | 0.108* |
| H16 | 0.4058 | 0.8964 | 0.8559 | 0.072* |
| H17 | 0.4016 | 0.7947 | 1.0042 | 0.094* |
| H18 | 0.2296 | 0.5714 | 1.0018 | 0.106* |
| H19 | 0.0603 | 0.4538 | 0.8546 | 0.088* |
| H20a | 0.4042 | 0.7770 | 0.5861 | 0.060* |
| H20b | 0.4817 | 0.7994 | 0.7018 | 0.060* |
| H21 | 0.3879 | 1.0336 | 0.5932 | 0.059* |
| H25a | 0.6590 | 0.9496 | 0.6126 | 0.061* |
| H25b | 0.6403 | 1.1174 | 0.5971 | 0.061* |
| H27 | 0.9342 | 1.1181 | 0.6546 | 0.064* |
| H28 | 1.1996 | 1.1626 | 0.7175 | 0.076* |
| H29 | 1.3025 | 1.1813 | 0.8877 | 0.086* |
| H30 | 1.1397 | 1.1641 | 0.9980 | 0.089* |
| H31 | 0.8735 | 1.1235 | 0.9370 | 0.073* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O14 | 0.0543 (9) | 0.0779 (11) | 0.0738 (11) | 0.0015 (7) | −0.0006 (7) | 0.0224 (8) |
| O15 | 0.0587 (10) | 0.1559 (19) | 0.0490 (9) | −0.0199 (10) | 0.0048 (7) | 0.0315 (10) |
| O22 | 0.0372 (7) | 0.0582 (8) | 0.0719 (9) | 0.0069 (6) | 0.0176 (6) | −0.0036 (6) |
| N10 | 0.0436 (8) | 0.0540 (9) | 0.0424 (8) | −0.0008 (7) | 0.0114 (6) | 0.0051 (6) |
| N13 | 0.0544 (10) | 0.0427 (9) | 0.0903 (13) | 0.0053 (7) | 0.0271 (9) | −0.0003 (9) |
| N23 | 0.0422 (9) | 0.0560 (9) | 0.0592 (10) | 0.0041 (7) | 0.0155 (7) | −0.0031 (7) |
| C1 | 0.0456 (10) | 0.0480 (10) | 0.0690 (13) | 0.0034 (8) | 0.0147 (9) | 0.0102 (9) |
| C2 | 0.0495 (12) | 0.1070 (19) | 0.0675 (14) | 0.0148 (12) | 0.0178 (10) | 0.0111 (13) |
| C3 | 0.0662 (16) | 0.125 (2) | 0.0872 (18) | 0.0373 (16) | 0.0352 (14) | 0.0422 (17) |
| C4 | 0.0687 (17) | 0.0772 (17) | 0.146 (3) | 0.0275 (14) | 0.0540 (18) | 0.0518 (18) |
| C5 | 0.0519 (14) | 0.0548 (14) | 0.171 (3) | −0.0052 (11) | 0.0279 (17) | 0.0121 (17) |
| C6 | 0.0554 (13) | 0.0520 (12) | 0.113 (2) | −0.0036 (10) | 0.0177 (13) | −0.0087 (12) |
| C7 | 0.0489 (11) | 0.0520 (11) | 0.0580 (11) | −0.0035 (8) | 0.0162 (9) | −0.0055 (9) |
| C8 | 0.0445 (10) | 0.0541 (11) | 0.0518 (11) | −0.0007 (8) | 0.0082 (8) | 0.0049 (8) |
| C9 | 0.0467 (11) | 0.0674 (12) | 0.0446 (10) | −0.0082 (9) | 0.0066 (8) | 0.0070 (9) |
| C11 | 0.0475 (10) | 0.0532 (10) | 0.0479 (10) | 0.0138 (8) | 0.0178 (8) | 0.0106 (8) |
| C12 | 0.0527 (11) | 0.0506 (11) | 0.0713 (13) | 0.0198 (9) | 0.0284 (10) | 0.0173 (9) |
| C16 | 0.0611 (12) | 0.0728 (13) | 0.0478 (11) | 0.0184 (10) | 0.0133 (9) | 0.0085 (9) |
| C17 | 0.0833 (17) | 0.113 (2) | 0.0503 (13) | 0.0401 (16) | 0.0175 (11) | 0.0187 (13) |
| C18 | 0.103 (2) | 0.115 (2) | 0.0788 (18) | 0.0539 (19) | 0.0466 (17) | 0.0552 (17) |
| C19 | 0.0775 (16) | 0.0741 (15) | 0.0921 (19) | 0.0343 (13) | 0.0428 (14) | 0.0422 (14) |
| C20 | 0.0439 (10) | 0.0563 (11) | 0.0471 (10) | 0.0031 (8) | 0.0152 (8) | 0.0016 (8) |
| C21 | 0.0375 (9) | 0.0577 (11) | 0.0511 (10) | 0.0055 (8) | 0.0104 (8) | 0.0083 (8) |
| C24 | 0.0401 (9) | 0.0447 (9) | 0.0519 (10) | 0.0050 (7) | 0.0147 (8) | 0.0056 (7) |
| C25 | 0.0389 (10) | 0.0609 (11) | 0.0508 (10) | 0.0030 (8) | 0.0136 (8) | 0.0092 (8) |
| C26 | 0.0398 (9) | 0.0439 (9) | 0.0531 (10) | 0.0051 (7) | 0.0110 (8) | 0.0069 (8) |
| C27 | 0.0437 (10) | 0.0561 (11) | 0.0585 (11) | 0.0078 (8) | 0.0141 (9) | 0.0046 (9) |
| C28 | 0.0406 (10) | 0.0675 (13) | 0.0827 (15) | 0.0112 (9) | 0.0182 (10) | 0.0046 (11) |
| C29 | 0.0412 (11) | 0.0707 (14) | 0.0983 (19) | 0.0137 (10) | 0.0022 (11) | 0.0102 (13) |
| C30 | 0.0646 (14) | 0.0812 (16) | 0.0663 (14) | 0.0123 (12) | −0.0075 (11) | 0.0163 (12) |
| C31 | 0.0546 (12) | 0.0712 (13) | 0.0562 (12) | 0.0088 (10) | 0.0104 (9) | 0.0155 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O14—C8 | 1.412 (3) | C11—C16 | 1.386 (3) |
| O14—H14 | 0.8200 | C12—C19 | 1.395 (3) |
| O15—C9 | 1.227 (3) | C16—C17 | 1.379 (4) |
| O22—N23 | 1.415 (2) | C16—H16 | 0.9300 |
| O22—C21 | 1.446 (2) | C17—C18 | 1.383 (5) |
| N10—C9 | 1.345 (2) | C17—H17 | 0.9300 |
| N10—C11 | 1.432 (2) | C18—C19 | 1.366 (4) |
| N10—C20 | 1.473 (2) | C18—H18 | 0.9300 |
| N13—C7 | 1.475 (3) | C19—H19 | 0.9300 |
| N13—C12 | 1.411 (3) | C20—C21 | 1.516 (3) |
| N13—H13 | 0.931 (19) | C20—H20a | 0.9700 |

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| N23—C24 | 1.280 (3) | C20—H20b | 0.9700 |
| C1—C2 | 1.390 (3) | C21—C25 | 1.523 (3) |
| C1—C6 | 1.376 (3) | C21—H21 | 0.9800 |
| C1—C7 | 1.516 (3) | C24—C25 | 1.497 (3) |
| C2—C3 | 1.377 (4) | C24—C26 | 1.470 (3) |
| C2—H2 | 0.9300 | C25—H25a | 0.9700 |
| C3—C4 | 1.363 (4) | C25—H25b | 0.9700 |
| C3—H3 | 0.9300 | C26—C27 | 1.395 (3) |
| C4—C5 | 1.363 (5) | C26—C31 | 1.388 (3) |
| C4—H4 | 0.9300 | C27—C28 | 1.378 (3) |
| C5—C6 | 1.404 (4) | C27—H27 | 0.9300 |
| C5—H5 | 0.9300 | C28—C29 | 1.364 (4) |
| C6—H6 | 0.9300 | C28—H28 | 0.9300 |
| C7—C8 | 1.529 (3) | C29—C30 | 1.385 (4) |
| C7—H7 | 0.9800 | C29—H29 | 0.9300 |
| C8—C9 | 1.522 (3) | C30—C31 | 1.381 (4) |
| C8—H8 | 0.9800 | C30—H30 | 0.9300 |
| C11—C12 | 1.396 (3) | C31—H31 | 0.9300 |

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| C8—O14—H14 | 109.00 | C17—C16—H16 | 120.00 |
| N23—O22—C21 | 108.51 (14) | C16—C17—C18 | 119.5 (2) |
| C9—N10—C11 | 122.14 (16) | C16—C17—H17 | 120.00 |
| C9—N10—C20 | 117.82 (15) | C18—C17—H17 | 120.00 |
| C11—N10—C20 | 119.62 (15) | C17—C18—C19 | 120.5 (3) |
| C7—N13—C12 | 117.66 (16) | C17—C18—H18 | 120.00 |
| C7—N13—H13 | 109.8 (17) | C19—C18—H18 | 120.00 |
| C12—N13—H13 | 110.1 (16) | C12—C19—C18 | 121.2 (3) |
| O22—N23—C24 | 108.88 (16) | C12—C19—H19 | 119.00 |
| C2—C1—C6 | 117.1 (2) | C18—C19—H19 | 119.00 |
| C2—C1—C7 | 122.4 (2) | N10—C20—C21 | 114.08 (15) |
| C6—C1—C7 | 120.5 (2) | N10—C20—H20a | 109.00 |
| C1—C2—C3 | 121.6 (2) | N10—C20—H20b | 109.00 |
| C1—C2—H2 | 119.00 | C21—C20—H20a | 109.00 |
| C3—C2—H2 | 119.00 | C21—C20—H20b | 109.00 |
| C2—C3—C4 | 120.8 (3) | H20a—C20—H20b | 108.00 |
| C2—C3—H3 | 120.00 | O22—C21—C20 | 109.67 (15) |
| C4—C3—H3 | 120.00 | O22—C21—C25 | 103.98 (14) |
| C3—C4—C5 | 119.1 (3) | O22—C21—H21 | 110.00 |
| C3—C4—H4 | 120.00 | C20—C21—C25 | 111.47 (15) |
| C5—C4—H4 | 120.00 | C20—C21—H21 | 111.00 |
| C4—C5—C6 | 120.5 (3) | C25—C21—H21 | 111.00 |
| C4—C5—H5 | 120.00 | N23—C24—C25 | 113.70 (17) |
| C6—C5—H5 | 120.00 | N23—C24—C26 | 120.91 (17) |
| C1—C6—C5 | 120.9 (3) | C25—C24—C26 | 125.39 (16) |
| C1—C6—H6 | 120.00 | C21—C25—C24 | 100.10 (15) |
| C5—C6—H6 | 120.00 | C21—C25—H25a | 112.00 |
| N13—C7—C1 | 113.45 (17) | C21—C25—H25b | 112.00 |
| N13—C7—C8 | 109.22 (16) | C24—C25—H25a | 112.00 |

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| N13—C7—H7 | 107.00 | C24—C25—H25b | 112.00 |
| C1—C7—C8 | 112.31 (15) | H25a—C25—H25b | 109.00 |
| C1—C7—H7 | 107.00 | C24—C26—C27 | 119.42 (17) |
| C8—C7—H7 | 107.00 | C24—C26—C31 | 121.77 (19) |
| O14—C8—C7 | 108.40 (16) | C27—C26—C31 | 118.82 (19) |
| O14—C8—C9 | 109.81 (16) | C26—C27—C28 | 120.3 (2) |
| O14—C8—H8 | 109.00 | C26—C27—H27 | 120.00 |
| C7—C8—C9 | 112.00 (15) | C28—C27—H27 | 120.00 |
| C7—C8—H8 | 109.00 | C27—C28—C29 | 120.5 (2) |
| C9—C8—H8 | 109.00 | C27—C28—H28 | 120.00 |
| O15—C9—N10 | 122.11 (18) | C29—C28—H28 | 120.00 |
| O15—C9—C8 | 119.30 (18) | C28—C29—C30 | 119.9 (2) |
| N10—C9—C8 | 118.48 (16) | C28—C29—H29 | 120.00 |
| N10—C11—C12 | 119.65 (17) | C30—C29—H29 | 120.00 |
| N10—C11—C16 | 119.64 (18) | C29—C30—C31 | 120.3 (2) |
| C12—C11—C16 | 120.59 (19) | C29—C30—H30 | 120.00 |
| N13—C12—C11 | 120.15 (19) | C31—C30—H30 | 120.00 |
| N13—C12—C19 | 121.85 (19) | C26—C31—C30 | 120.2 (2) |
| C11—C12—C19 | 117.9 (2) | C26—C31—H31 | 120.00 |
| C11—C16—C17 | 120.3 (2) | C30—C31—H31 | 120.00 |
| C11—C16—H16 | 120.00 | | |

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| O14—C8—C9—O15 | −15.0 (3) | C7—C8—C9—O15 | 105.5 (2) |
| O14—C8—C9—N10 | 161.30 (17) | C7—C8—C9—N10 | −78.2 (2) |
| O22—N23—C24—C25 | 0.4 (2) | C9—N10—C11—C12 | 42.8 (3) |
| O22—N23—C24—C26 | 179.36 (16) | C9—N10—C11—C16 | −141.1 (2) |
| O22—C21—C25—C24 | 20.06 (17) | C9—N10—C20—C21 | 74.2 (2) |
| N10—C11—C12—N13 | −1.1 (3) | C11—N10—C9—O15 | −172.6 (2) |
| N10—C11—C12—C19 | 175.89 (19) | C11—N10—C9—C8 | 11.3 (3) |
| N10—C11—C16—C17 | −174.9 (2) | C11—N10—C20—C21 | −113.19 (18) |
| N10—C20—C21—O22 | 58.2 (2) | C11—C12—C19—C18 | −1.2 (4) |
| N10—C20—C21—C25 | 172.78 (15) | C11—C16—C17—C18 | −0.7 (4) |
| N13—C7—C8—O14 | 162.39 (16) | C12—N13—C7—C1 | −79.9 (2) |
| N13—C7—C8—C9 | 41.1 (2) | C12—N13—C7—C8 | 46.2 (2) |
| N13—C12—C19—C18 | 175.7 (3) | C12—C11—C16—C17 | 1.1 (4) |
| N23—O22—C21—C20 | 97.71 (17) | C16—C11—C12—N13 | −177.1 (2) |
| N23—O22—C21—C25 | −21.61 (18) | C16—C11—C12—C19 | −0.1 (3) |
| N23—C24—C25—C21 | −13.3 (2) | C16—C17—C18—C19 | −0.7 (5) |
| N23—C24—C26—C27 | −163.20 (18) | C17—C18—C19—C12 | 1.7 (5) |
| N23—C24—C26—C31 | 17.0 (3) | C20—N10—C9—O15 | −0.1 (3) |
| C1—C2—C3—C4 | −0.8 (5) | C20—N10—C9—C8 | −176.28 (15) |
| C1—C7—C8—O14 | −70.8 (2) | C20—N10—C11—C12 | −129.51 (19) |
| C1—C7—C8—C9 | 167.85 (17) | C20—N10—C11—C16 | 46.5 (3) |
| C2—C1—C6—C5 | −1.8 (4) | C20—C21—C25—C24 | −98.02 (17) |
| C2—C1—C7—N13 | 67.0 (3) | C21—O22—N23—C24 | 13.9 (2) |
| C2—C1—C7—C8 | −57.4 (3) | C24—C26—C27—C28 | 179.47 (19) |
| C2—C3—C4—C5 | −1.1 (5) | C24—C26—C31—C30 | 180.0 (2) |
| C3—C4—C5—C6 | 1.5 (4) | C25—C24—C26—C27 | 15.6 (3) |

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| C4—C5—C6—C1 | 0.0 (4) | C25—C24—C26—C31 | −164.2 (2) |
| C6—C1—C2—C3 | 2.2 (4) | C26—C24—C25—C21 | 167.78 (17) |
| C6—C1—C7—N13 | −111.5 (2) | C26—C27—C28—C29 | 1.2 (4) |
| C6—C1—C7—C8 | 124.0 (2) | C27—C26—C31—C30 | 0.1 (3) |
| C7—N13—C12—C11 | −73.2 (2) | C27—C28—C29—C30 | −1.1 (4) |
| C7—N13—C12—C19 | 110.0 (2) | C28—C29—C30—C31 | 0.5 (4) |
| C7—C1—C2—C3 | −176.4 (2) | C29—C30—C31—C26 | 0.0 (4) |
| C7—C1—C6—C5 | 176.8 (2) | C31—C26—C27—C28 | −0.7 (3) |

*Hydrogen-bond geometry (Å, º)*

Cg4 is the centroid of the C26–C31 ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O14—H14···O15 | 0.82 | 2.14 | 2.632 (3) | 118 |
| C16—H16···O22 | 0.93 | 2.44 | 3.206 (3) | 140 |
| C25—H25*a*···O14$^{i}$ | 0.97 | 2.59 | 3.493 (3) | 154 |
| C27—H27···O15$^{ii}$ | 0.93 | 2.40 | 3.302 (3) | 162 |
| C28—H28···O22$^{i}$ | 0.93 | 2.52 | 3.152 (2) | 125 |
| C19—H19···*Cg*4$^{iii}$ | 0.93 | 2.78 | 3.534 (3) | 139 |
| N13—H13···*Cg*4$^{iii}$ | 0.93 (2) | 2.77 (2) | 3.671 (3) | 164 |

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