IUCrData

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# (3*S*,4*S*)-4-Phenyl-1,5-bis(prop-2-en-1-yl)-3-(prop-2-en-1-yloxy)-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-2-one

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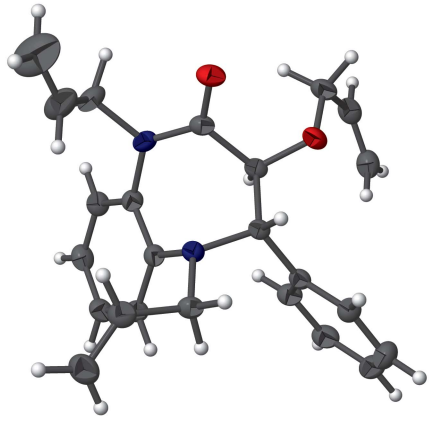
**Keywords:** crystal structure; benzodiazepine derivative; hydrogen bond.

**CCDC reference**: 1494818

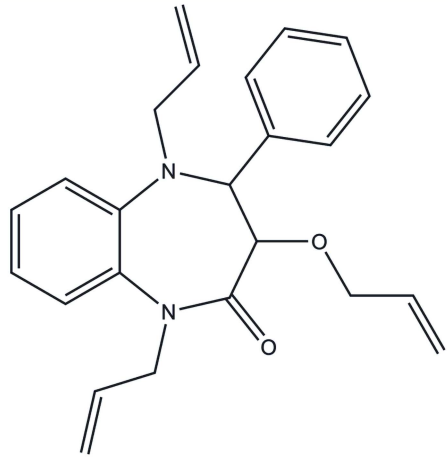
**Structural data**: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{24}H_{26}N_2O_2$, the dihedral angle between the benzene rings is 45.69 (7)°. In the crystal, the molecules form helical supramolecular chains running parallel to the *b* axis *via* weak C—H···O hydrogen bonds.

**3D view**



**Chemical scheme**




## Structure description

1,5-Benzodiazepine derivatives have been used as therapeutics for viral infection and cardiovascular disorder (Jacob *et al.*, 2011; Maleki *et al.*, 2014). They are active against peptide hormones (Werner *et al.*, 1990) and potassium blockers (Claremon *et al.*, 1996). They are also employed as intermediates for the synthesis of several heterocyclic compounds (Minnih *et al.*, 2014). As part of our studies in this area, we now describe the synthesis and structure of the title compound.

In the title molecule (Fig. 1), the dihedral angle between the C1–C6 and C10–C15 rings is 45.69 (7)°. Analysis of the conformation of the seven-membered ring yielded puckering parameters $Q(2)$ = 1.034 (2) Å, $\varphi(2)$ = 227.0 (1)°, $Q(3)$ = 0.174 (2) Å and $\varphi(3)$ = 1.3 (6)°. In the crystal, the molecules form supramolecular helical chains parallel to the *b* axis through C22—H22*A*···O1($-x+1$, $y+\frac{1}{2}$, $-z$) hydrogen bonds (Table 1 and Fig. 2).

## Synthesis and crystallization

To a solution of 3-hydroxy-4-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one (1 g, 3.5 mmol) in DMF (20 ml) were added allyl bromide (0.5 g. 10.5 mmol), potassium carbonate (1 g, 7.4 mmol) and a catalytic quantity of tetra-*n*-butyl ammonium bromide.

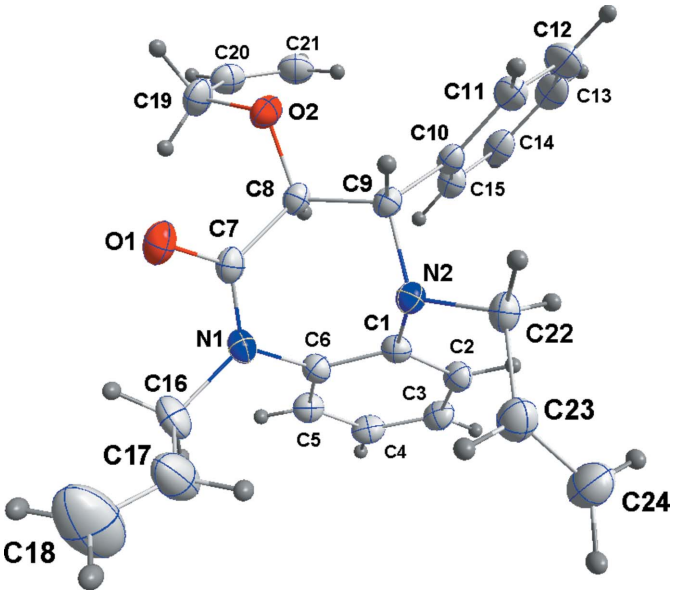



**Figure 1**
The title molecule with labeling scheme and 50% probability ellipsoids.

The mixture was stirred at room temperature for 24 h. The solution was filtered and the solvent removed under reduced pressure. The residue was recrystallized from ethanol solution to afford the compound as colorless crystals.

## Refinement

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


## Acknowledgements

The support of NSF–MRI Grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

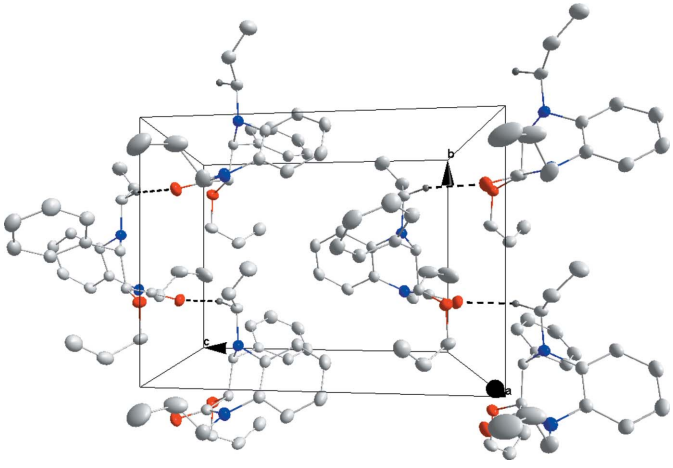




**Figure 2**
Packing viewed along the *a* axis with C—H···O hydrogen bonds shown as dashed lines.

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C22—H22$A$···O1$^{i}$ | 0.99 | 2.41 | 3.378 (2) | 165 |

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z$.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{24}H_{26}N_2O_2$ |
| $M_r$ | 374.47 |
| Crystal system, space group | Monoclinic, $P2_1$ |
| Temperature (K) | 150 |
| $a, b, c$ (Å) | 9.6138 (2), 8.8891 (2), 11.7292 (3) |
| $\beta$ (°) | 97.127 (1) |
| $V$ (Å$^3$) | 994.61 (4) |
| $Z$ | 2 |
| Radiation type | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.63 |
| Crystal size (mm) | 0.21 × 0.15 × 0.14 |
| Data collection | |
| Diffractometer | Bruker D8 VENTURE PHOTON 100 CMOS |
| Absorption correction | Multi-scan (*SADABS*; Bruker, 2016) |
| $T_{min}$, $T_{max}$ | 0.84, 0.90 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 9888, 3881, 3744 |
| $R_{int}$ | 0.029 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.624 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.030, 0.075, 1.05 |
| No. of reflections | 3881 |
| No. of parameters | 254 |
| No. of restraints | 1 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.18, −0.21 |
| Absolute structure | Flack $x$ determined using 1608 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) |
| Absolute structure parameter | 0.00 (8) |

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

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# full crystallographic data

*IUCrData* (2016). **1**, x161175 [https://doi.org/10.1107/S2414314616011755]

## (3*S*,4*S*)-4-Phenyl-1,5-bis(prop-2-en-1-yl)-3-(prop-2-en-1-yloxy)-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-2-one

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**(3*S*,4*S*)-4-Phenyl-1,5-bis(prop-2-en-1-yl)-3-(prop-2-en-1-yloxy)-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-2-one**

*Crystal data*

$C_{24}H_{26}N_2O_2$
$M_r$ = 374.47
Monoclinic, $P2_1$
$a$ = 9.6138 (2) Å
$b$ = 8.8891 (2) Å
$c$ = 11.7292 (3) Å
$\beta$ = 97.127 (1)°
$V$ = 994.61 (4) Å$^3$
$Z$ = 2
$F(000)$ = 400
$D_x$ = 1.250 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54178 Å
Cell parameters from 9162 reflections
$\theta$ = 3.8–74.3°
$\mu$ = 0.63 mm$^{-1}$
$T$ = 150 K
Block, colourless
0.21 × 0.15 × 0.14 mm

*Data collection*

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC I$\mu$S micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
$T_{min}$ = 0.84, $T_{max}$ = 0.90
9888 measured reflections
3881 independent reflections
3744 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029
$\theta_{max}$ = 74.3°, $\theta_{min}$ = 3.8°
$h$ = −11→10
$k$ = −11→11
$l$ = −14→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.075
$S$ = 1.05
3881 reflections
254 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0347P)^2 + 0.1749P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$
Extinction correction: SHELXL2014 (Sheldrick 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0076 (8)
Absolute structure: Flack $x$ determined using 1608 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.00 (8)

*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.

**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 $F^2 > 2\text{sigma}(F^2)$ 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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 1.00 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.36677 (15) | 0.28288 (17) | 0.02270 (11) | 0.0310 (3) |
| O2 | 0.64430 (14) | 0.29097 (15) | 0.10026 (11) | 0.0274 (3) |
| N1 | 0.30144 (16) | 0.32889 (18) | 0.19803 (13) | 0.0256 (3) |
| N2 | 0.45776 (15) | 0.59426 (16) | 0.22157 (12) | 0.0216 (3) |
| C1 | 0.40506 (18) | 0.5341 (2) | 0.31988 (15) | 0.0223 (4) |
| C2 | 0.4206 (2) | 0.6060 (2) | 0.42694 (15) | 0.0261 (4) |
| H2 | 0.4703 | 0.6984 | 0.4363 | 0.031* |
| C3 | 0.3642 (2) | 0.5435 (2) | 0.51948 (16) | 0.0302 (4) |
| H3 | 0.3751 | 0.5938 | 0.5915 | 0.036* |
| C4 | 0.2924 (2) | 0.4087 (2) | 0.50753 (17) | 0.0305 (4) |
| H4 | 0.2543 | 0.3662 | 0.5712 | 0.037* |
| C5 | 0.2763 (2) | 0.3357 (2) | 0.40224 (16) | 0.0283 (4) |
| H5 | 0.2285 | 0.2421 | 0.3943 | 0.034* |
| C6 | 0.32954 (19) | 0.3986 (2) | 0.30810 (16) | 0.0239 (4) |
| C7 | 0.39812 (19) | 0.31974 (19) | 0.12316 (15) | 0.0241 (4) |
| C8 | 0.55078 (19) | 0.3467 (2) | 0.17397 (14) | 0.0225 (4) |
| H8 | 0.5695 | 0.2965 | 0.2506 | 0.027* |
| C9 | 0.58168 (18) | 0.51561 (19) | 0.18692 (14) | 0.0204 (3) |
| H9 | 0.5942 | 0.5537 | 0.1086 | 0.025* |
| C10 | 0.71810 (18) | 0.5486 (2) | 0.26366 (15) | 0.0231 (4) |
| C11 | 0.8169 (2) | 0.6427 (2) | 0.22330 (17) | 0.0289 (4) |
| H11 | 0.7991 | 0.6838 | 0.1481 | 0.035* |
| C12 | 0.9414 (2) | 0.6774 (3) | 0.2916 (2) | 0.0380 (5) |
| H12 | 1.0070 | 0.7434 | 0.2635 | 0.046* |
| C13 | 0.9702 (2) | 0.6164 (3) | 0.4004 (2) | 0.0387 (5) |
| H13 | 1.0559 | 0.6390 | 0.4467 | 0.046* |
| C14 | 0.8730 (2) | 0.5220 (3) | 0.44140 (18) | 0.0345 (5) |
| H14 | 0.8926 | 0.4792 | 0.5159 | 0.041* |
| C15 | 0.7470 (2) | 0.4894 (2) | 0.37439 (16) | 0.0285 (4) |
| H15 | 0.6802 | 0.4264 | 0.4041 | 0.034* |
| C16 | 0.1552 (2) | 0.2936 (3) | 0.15412 (18) | 0.0337 (5) |
| H16A | 0.1036 | 0.2701 | 0.2199 | 0.040* |
| H16B | 0.1536 | 0.2025 | 0.1054 | 0.040* |
| C17 | 0.0813 (2) | 0.4166 (3) | 0.0863 (3) | 0.0510 (7) |

| | | | | |
|---|---|---|---|---|
| H17 | 0.1021 | 0.5168 | 0.1109 | 0.061* |
| C18 | −0.0070 (4) | 0.3989 (4) | −0.0016 (4) | 0.0751 (10) |
| H18A | −0.0306 | 0.3004 | −0.0290 | 0.090* |
| H18B | −0.0498 | 0.4840 | −0.0404 | 0.090* |
| C19 | 0.6504 (2) | 0.1307 (2) | 0.09502 (18) | 0.0312 (4) |
| H19A | 0.6713 | 0.1006 | 0.0177 | 0.037* |
| H19B | 0.5572 | 0.0893 | 0.1055 | 0.037* |
| C20 | 0.7576 (2) | 0.0632 (2) | 0.18291 (18) | 0.0332 (4) |
| H20 | 0.7602 | −0.0435 | 0.1873 | 0.040* |
| C21 | 0.8487 (2) | 0.1365 (3) | 0.25474 (19) | 0.0368 (5) |
| H21A | 0.8503 | 0.2434 | 0.2538 | 0.044* |
| H21B | 0.9131 | 0.0828 | 0.3078 | 0.044* |
| C22 | 0.4695 (2) | 0.75892 (19) | 0.21535 (16) | 0.0253 (4) |
| H22A | 0.5147 | 0.7867 | 0.1469 | 0.030* |
| H22B | 0.5295 | 0.7961 | 0.2843 | 0.030* |
| C23 | 0.3291 (2) | 0.8316 (2) | 0.20827 (17) | 0.0316 (4) |
| H23 | 0.2597 | 0.7985 | 0.1488 | 0.038* |
| C24 | 0.2929 (3) | 0.9369 (3) | 0.2767 (2) | 0.0405 (5) |
| H24A | 0.3589 | 0.9734 | 0.3373 | 0.049* |
| H24B | 0.2006 | 0.9769 | 0.2657 | 0.049* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0383 (8) | 0.0314 (7) | 0.0217 (6) | 0.0001 (6) | −0.0022 (5) | −0.0053 (5) |
| O2 | 0.0341 (7) | 0.0220 (6) | 0.0272 (6) | 0.0019 (5) | 0.0080 (5) | −0.0026 (5) |
| N1 | 0.0256 (8) | 0.0280 (8) | 0.0224 (7) | −0.0060 (6) | 0.0001 (6) | −0.0007 (6) |
| N2 | 0.0243 (7) | 0.0201 (7) | 0.0205 (7) | 0.0002 (6) | 0.0029 (5) | 0.0004 (6) |
| C1 | 0.0214 (8) | 0.0247 (8) | 0.0205 (8) | 0.0013 (7) | 0.0017 (6) | 0.0014 (7) |
| C2 | 0.0273 (9) | 0.0274 (10) | 0.0231 (9) | 0.0005 (7) | 0.0008 (7) | −0.0016 (7) |
| C3 | 0.0335 (10) | 0.0356 (11) | 0.0214 (9) | 0.0044 (8) | 0.0030 (7) | −0.0020 (8) |
| C4 | 0.0330 (10) | 0.0345 (10) | 0.0249 (9) | 0.0054 (8) | 0.0069 (7) | 0.0067 (8) |
| C5 | 0.0296 (10) | 0.0275 (9) | 0.0279 (9) | 0.0014 (8) | 0.0048 (7) | 0.0051 (8) |
| C6 | 0.0236 (8) | 0.0255 (9) | 0.0223 (9) | −0.0002 (7) | 0.0016 (6) | 0.0010 (7) |
| C7 | 0.0296 (9) | 0.0187 (8) | 0.0232 (8) | −0.0003 (7) | 0.0004 (7) | −0.0006 (7) |
| C8 | 0.0276 (9) | 0.0208 (8) | 0.0190 (8) | 0.0004 (7) | 0.0022 (6) | −0.0008 (6) |
| C9 | 0.0238 (8) | 0.0196 (8) | 0.0178 (8) | 0.0006 (7) | 0.0028 (6) | 0.0002 (6) |
| C10 | 0.0241 (9) | 0.0213 (8) | 0.0235 (8) | 0.0031 (7) | 0.0014 (6) | −0.0024 (7) |
| C11 | 0.0268 (9) | 0.0300 (10) | 0.0300 (10) | −0.0010 (8) | 0.0036 (7) | −0.0009 (8) |
| C12 | 0.0261 (10) | 0.0431 (12) | 0.0446 (12) | −0.0044 (9) | 0.0035 (9) | −0.0022 (10) |
| C13 | 0.0274 (10) | 0.0430 (12) | 0.0426 (12) | 0.0030 (9) | −0.0083 (8) | −0.0104 (10) |
| C14 | 0.0374 (11) | 0.0343 (10) | 0.0291 (10) | 0.0075 (9) | −0.0066 (8) | −0.0032 (8) |
| C15 | 0.0322 (10) | 0.0272 (10) | 0.0247 (9) | 0.0025 (8) | −0.0016 (8) | −0.0001 (7) |
| C16 | 0.0285 (10) | 0.0393 (11) | 0.0317 (9) | −0.0131 (9) | −0.0026 (8) | 0.0041 (9) |
| C17 | 0.0320 (12) | 0.0336 (12) | 0.083 (2) | −0.0002 (10) | −0.0099 (12) | 0.0005 (12) |
| C18 | 0.0630 (18) | 0.0597 (19) | 0.092 (2) | 0.0049 (16) | −0.0326 (17) | 0.0235 (17) |
| C19 | 0.0375 (11) | 0.0224 (9) | 0.0334 (10) | 0.0036 (8) | 0.0030 (8) | −0.0060 (7) |
| C20 | 0.0363 (10) | 0.0280 (10) | 0.0366 (11) | 0.0059 (8) | 0.0096 (8) | 0.0040 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C21 | 0.0321 (10) | 0.0410 (12) | 0.0378 (11) | 0.0059 (9) | 0.0063 (8) | 0.0041 (9) |
| C22 | 0.0301 (9) | 0.0188 (8) | 0.0267 (9) | −0.0014 (7) | 0.0022 (7) | 0.0000 (7) |
| C23 | 0.0324 (10) | 0.0271 (10) | 0.0340 (10) | 0.0037 (8) | −0.0014 (8) | 0.0005 (8) |
| C24 | 0.0416 (12) | 0.0348 (12) | 0.0447 (13) | 0.0094 (9) | 0.0044 (10) | −0.0033 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C7 | 1.224 (2) | C12—C13 | 1.382 (3) |
| O2—C8 | 1.412 (2) | C12—H12 | 0.9500 |
| O2—C19 | 1.427 (2) | C13—C14 | 1.386 (3) |
| N1—C7 | 1.358 (2) | C13—H13 | 0.9500 |
| N1—C6 | 1.427 (2) | C14—C15 | 1.390 (3) |
| N1—C16 | 1.470 (2) | C14—H14 | 0.9500 |
| N2—C1 | 1.420 (2) | C15—H15 | 0.9500 |
| N2—C22 | 1.471 (2) | C16—C17 | 1.480 (3) |
| N2—C9 | 1.481 (2) | C16—H16A | 0.9900 |
| C1—C2 | 1.400 (3) | C16—H16B | 0.9900 |
| C1—C6 | 1.404 (3) | C17—C18 | 1.262 (4) |
| C2—C3 | 1.388 (3) | C17—H17 | 0.9500 |
| C2—H2 | 0.9500 | C18—H18A | 0.9500 |
| C3—C4 | 1.381 (3) | C18—H18B | 0.9500 |
| C3—H3 | 0.9500 | C19—C20 | 1.491 (3) |
| C4—C5 | 1.387 (3) | C19—H19A | 0.9900 |
| C4—H4 | 0.9500 | C19—H19B | 0.9900 |
| C5—C6 | 1.391 (3) | C20—C21 | 1.311 (3) |
| C5—H5 | 0.9500 | C20—H20 | 0.9500 |
| C7—C8 | 1.533 (2) | C21—H21A | 0.9500 |
| C8—C9 | 1.534 (2) | C21—H21B | 0.9500 |
| C8—H8 | 1.0000 | C22—C23 | 1.490 (3) |
| C9—C10 | 1.524 (2) | C22—H22A | 0.9900 |
| C9—H9 | 1.0000 | C22—H22B | 0.9900 |
| C10—C11 | 1.392 (3) | C23—C24 | 1.308 (3) |
| C10—C15 | 1.397 (3) | C23—H23 | 0.9500 |
| C11—C12 | 1.390 (3) | C24—H24A | 0.9500 |
| C11—H11 | 0.9500 | C24—H24B | 0.9500 |
| | | | |
| C8—O2—C19 | 114.15 (15) | C13—C12—H12 | 119.8 |
| C7—N1—C6 | 122.88 (15) | C11—C12—H12 | 119.8 |
| C7—N1—C16 | 117.54 (15) | C12—C13—C14 | 119.42 (19) |
| C6—N1—C16 | 118.35 (16) | C12—C13—H13 | 120.3 |
| C1—N2—C22 | 116.90 (15) | C14—C13—H13 | 120.3 |
| C1—N2—C9 | 115.16 (14) | C13—C14—C15 | 120.5 (2) |
| C22—N2—C9 | 112.75 (14) | C13—C14—H14 | 119.7 |
| C2—C1—C6 | 118.43 (16) | C15—C14—H14 | 119.7 |
| C2—C1—N2 | 123.16 (17) | C14—C15—C10 | 120.37 (19) |
| C6—C1—N2 | 118.37 (16) | C14—C15—H15 | 119.8 |
| C3—C2—C1 | 120.67 (18) | C10—C15—H15 | 119.8 |
| C3—C2—H2 | 119.7 | N1—C16—C17 | 113.90 (18) |

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| C1—C2—H2 | 119.7 | N1—C16—H16A | 108.8 |
| C4—C3—C2 | 120.42 (18) | C17—C16—H16A | 108.8 |
| C4—C3—H3 | 119.8 | N1—C16—H16B | 108.8 |
| C2—C3—H3 | 119.8 | C17—C16—H16B | 108.8 |
| C3—C4—C5 | 119.72 (19) | H16A—C16—H16B | 107.7 |
| C3—C4—H4 | 120.1 | C18—C17—C16 | 125.2 (3) |
| C5—C4—H4 | 120.1 | C18—C17—H17 | 117.4 |
| C4—C5—C6 | 120.49 (19) | C16—C17—H17 | 117.4 |
| C4—C5—H5 | 119.8 | C17—C18—H18A | 120.0 |
| C6—C5—H5 | 119.8 | C17—C18—H18B | 120.0 |
| C5—C6—C1 | 120.23 (17) | H18A—C18—H18B | 120.0 |
| C5—C6—N1 | 119.76 (17) | O2—C19—C20 | 113.60 (17) |
| C1—C6—N1 | 119.94 (16) | O2—C19—H19A | 108.8 |
| O1—C7—N1 | 122.14 (17) | C20—C19—H19A | 108.8 |
| O1—C7—C8 | 121.71 (17) | O2—C19—H19B | 108.8 |
| N1—C7—C8 | 116.00 (15) | C20—C19—H19B | 108.8 |
| O2—C8—C7 | 111.01 (14) | H19A—C19—H19B | 107.7 |
| O2—C8—C9 | 105.90 (14) | C21—C20—C19 | 126.4 (2) |
| C7—C8—C9 | 110.85 (14) | C21—C20—H20 | 116.8 |
| O2—C8—H8 | 109.7 | C19—C20—H20 | 116.8 |
| C7—C8—H8 | 109.7 | C20—C21—H21A | 120.0 |
| C9—C8—H8 | 109.7 | C20—C21—H21B | 120.0 |
| N2—C9—C10 | 114.18 (14) | H21A—C21—H21B | 120.0 |
| N2—C9—C8 | 109.67 (14) | N2—C22—C23 | 111.07 (16) |
| C10—C9—C8 | 112.88 (14) | N2—C22—H22A | 109.4 |
| N2—C9—H9 | 106.5 | C23—C22—H22A | 109.4 |
| C10—C9—H9 | 106.5 | N2—C22—H22B | 109.4 |
| C8—C9—H9 | 106.5 | C23—C22—H22B | 109.4 |
| C11—C10—C15 | 118.51 (17) | H22A—C22—H22B | 108.0 |
| C11—C10—C9 | 119.24 (16) | C24—C23—C22 | 125.8 (2) |
| C15—C10—C9 | 122.24 (16) | C24—C23—H23 | 117.1 |
| C12—C11—C10 | 120.82 (19) | C22—C23—H23 | 117.1 |
| C12—C11—H11 | 119.6 | C23—C24—H24A | 120.0 |
| C10—C11—H11 | 119.6 | C23—C24—H24B | 120.0 |
| C13—C12—C11 | 120.3 (2) | H24A—C24—H24B | 120.0 |

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| C22—N2—C1—C2 | −28.3 (2) | C1—N2—C9—C10 | −75.02 (19) |
| C9—N2—C1—C2 | 107.57 (19) | C22—N2—C9—C10 | 62.63 (19) |
| C22—N2—C1—C6 | 149.46 (16) | C1—N2—C9—C8 | 52.80 (19) |
| C9—N2—C1—C6 | −74.7 (2) | C22—N2—C9—C8 | −169.56 (14) |
| C6—C1—C2—C3 | 0.8 (3) | O2—C8—C9—N2 | 157.45 (13) |
| N2—C1—C2—C3 | 178.56 (16) | C7—C8—C9—N2 | 36.95 (18) |
| C1—C2—C3—C4 | 0.4 (3) | O2—C8—C9—C10 | −74.02 (17) |
| C2—C3—C4—C5 | −0.2 (3) | C7—C8—C9—C10 | 165.48 (14) |
| C3—C4—C5—C6 | −1.2 (3) | N2—C9—C10—C11 | −105.72 (18) |
| C4—C5—C6—C1 | 2.5 (3) | C8—C9—C10—C11 | 128.12 (18) |
| C4—C5—C6—N1 | −174.51 (17) | N2—C9—C10—C15 | 73.3 (2) |
| C2—C1—C6—C5 | −2.2 (3) | C8—C9—C10—C15 | −52.8 (2) |

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| N2—C1—C6—C5 | 179.92 (16) | C15—C10—C11—C12 | −0.2 (3) |
| C2—C1—C6—N1 | 174.72 (16) | C9—C10—C11—C12 | 178.87 (18) |
| N2—C1—C6—N1 | −3.1 (2) | C10—C11—C12—C13 | 1.2 (3) |
| C7—N1—C6—C5 | −141.28 (18) | C11—C12—C13—C14 | −0.9 (3) |
| C16—N1—C6—C5 | 51.7 (2) | C12—C13—C14—C15 | −0.4 (3) |
| C7—N1—C6—C1 | 41.7 (3) | C13—C14—C15—C10 | 1.5 (3) |
| C16—N1—C6—C1 | −125.30 (19) | C11—C10—C15—C14 | −1.2 (3) |
| C6—N1—C7—O1 | −167.56 (17) | C9—C10—C15—C14 | 179.80 (18) |
| C16—N1—C7—O1 | −0.4 (3) | C7—N1—C16—C17 | −77.9 (3) |
| C6—N1—C7—C8 | 16.9 (2) | C6—N1—C16—C17 | 89.8 (2) |
| C16—N1—C7—C8 | −175.97 (16) | N1—C16—C17—C18 | 143.0 (3) |
| C19—O2—C8—C7 | −70.99 (19) | C8—O2—C19—C20 | −89.5 (2) |
| C19—O2—C8—C9 | 168.62 (14) | O2—C19—C20—C21 | −5.3 (3) |
| O1—C7—C8—O2 | −13.5 (2) | C1—N2—C22—C23 | −63.9 (2) |
| N1—C7—C8—O2 | 162.12 (15) | C9—N2—C22—C23 | 159.25 (15) |
| O1—C7—C8—C9 | 103.95 (19) | N2—C22—C23—C24 | 125.9 (2) |
| N1—C7—C8—C9 | −80.48 (19) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C22—H22*A*···O1$^{i}$ | 0.99 | 2.41 | 3.378 (2) | 165 |

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*.