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# 11-Hydroxy-9-[1-(4-methylphenyl)-4-oxo-3-phenylazetidin-2-yl]-18-oxo-10-oxa-2-azapentacyclo-[9.7.0.0$^{1,8}$.0$^{2,6}$.0$^{12,17}$]octadeca-12,14,16-triene-8-carbonitrile

**Sivasubramanian Suhitha,[a] Thothadri Srinivasan,[a] Raju Rajesh,[b] Raghavachary Raghunathan[b] and Devadasan Velmurugan[a]***

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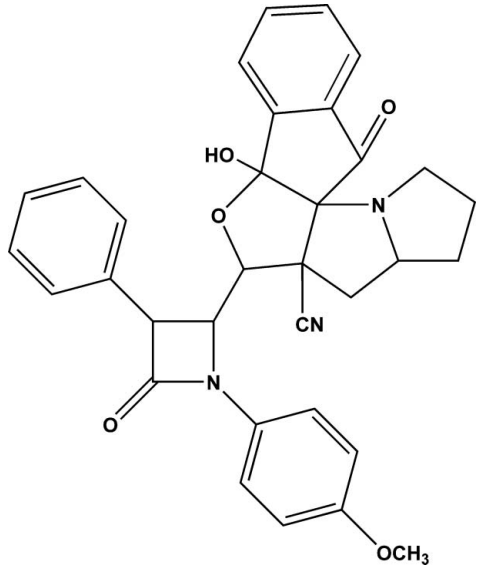
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.045; $wR$ factor = 0.123; data-to-parameter ratio = 17.1.

In the title compound, $C_{33}H_{29}N_3O_5$, the four-membered ring of the $\beta$-lactam fragment is essentially planar (r.m.s. deviation = 0.0122 Å), with the carbonyl O atom displaced from this ring by 0.856 (9) Å. The mean planes of the methoxyphenyl and phenyl rings are inclined at dihedral angles 85.10 (7) and 21.56 (14)°, respectively, with respect to the mean plane of the four-membered ring. The pyrrolidine rings adopt envelope conformations with C atoms lying 0.535 (4) and 0.519 (4) Å out of the planes formed by the remaining ring atoms. The furan ring also adopts an envelope conformation with a C atom 0.560 (3) Å out of the plane formed by the remaining ring atoms. The nine-membered indene ring is almost planar (r.m.s. deviation = 0.0240 Å), with the carbonyl O atom displaced by 0.145 (3) Å from this ring. The molecular structure is stabilized by a strong intramolecular O—H···N hydrogen bond and the crystal structure is consolidated by C—H···O hydrogen bonds.

## Related literature

For general background to $\beta$-lactams, see: Brakhage (1998). For a related structure, see: Sundaramoorthy *et al.* (2012).




## Experimental

### Crystal data

$C_{33}H_{29}N_3O_5$
$M_r$ = 547.59
Orthorhombic, $P2_12_12_1$
$a$ = 10.2874 (16) Å
$b$ = 14.138 (3) Å
$c$ = 18.866 (3) Å
$V$ = 2743.9 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 K
0.30 × 0.25 × 0.20 mm

### Data collection

Bruker SMART APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.973, $T_{max}$ = 0.982
14810 measured reflections
6406 independent reflections
4551 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.123
$S$ = 1.00
6406 reflections
375 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C10—H10···O2$^{i}$ | 0.98 | 2.43 | 3.337 (3) | 154 |
| C13—H13$A$···O2$^{i}$ | 0.97 | 2.41 | 3.321 (3) | 156 |
| C29—H29···O4$^{i}$ | 0.93 | 2.55 | 3.228 (3) | 130 |
| O4—H4$A$···N2 | 0.93 (3) | 1.84 (3) | 2.602 (3) | 137 (3) |

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

The authors thank the TBI X-ray facility, CAS in Crystallography and Biophysics, University of Madras, India, for the data collection and TS thanks the DST for an Inspire fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2596).

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# supporting information

*Acta Cryst.* (2012). E**68**, o3290–o3291 [doi:10.1107/S1600536812044479]

# 11-Hydroxy-9-[1-(4-methylphenyl)-4-oxo-3-phenylazetidin-2-yl]-18-oxo-10-oxa-2-azapentacyclo[9.7.0.0$^{1,8}$.0$^{2,6}$.0$^{12,17}$]octadeca-12,14,16-triene-8-carbonitrile

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## S1. Comment

The most commonly used *β*-lactam antibiotics for the therapy of infectious diseases are penicillin and cephalosporin (Brakhage, 1998). In view of potential applications, the crystal structure determination of the titled *β*-lactam derivative was carried out that is reported in this artiucle.

In the title compound (Fig. 1), the four membered ring of the *β*-lactam fragment (N1/C8–C10) is essentially planar (rmsd = 0.0122 Å) with O1 displaced from this ring by 0.856 (9) Å. The mean-planes of the benzene rings C2–C7 and C27–C32 are inclined at dihedral angles 85.10 (7) and 21.56 (14)°, respectively, with respect to the mean-plane of the four membered ring (N1/C8–C10).

The pyrrolidine rings (N1/C14–C17) and (N2/C12–C14/C18) adopt C16- and C13-envelope conformations with C16 and C13 atoms lying 0.535 (4) and 0.519 (4) Å, respectively, out of the planes formed by the remaining ring atoms. The furan ring (O3/C11/C12/C18/C19) also adopts a C11-envelope conformation with C11 atom 0.560 (3) Å out of the plane formed by the remaining ring atoms. The nine membered indene ring (C18–C26) is almost planar (rmsd = 0.0240 Å) with O5 displaced by 0.145 (3)Å from this ring.

The molecular structure of the title compound is stabilized by a strong intramolecular hydrogen bond O4—H4A···N2 (Table 1). The crystal structure is consolidated by intermolecular C—H···O hydrogen bonds (Tab. 1 & Fig. 2).

## S2. Experimental

A reaction mixture of 2-(hydroxy(1-(4-methoxyphenyl)-4-oxo-3-phenylazetidin -2-yl)methyl)acrylonitrile (1.0 mmol), ninhydrine (1.1 mmol) and proline (1.1 mmol) was refluxed in methanol (20 ml) until completion of the reaction was evidenced by TLC analysis. After completion of the reaction the solvent was evaporated under reduced pressure. The crude reaction mixture was dissolved in dichloromethane (2 x 50 ml) and washed with water followed by brine solution. The organic layer was separated and dried over sodium sulfate. After filteration and evaporation of the organic solvent was carried out under reduced pressure. The product was separated by column chromatography using hexane and ethyl acetate (4:6) as an eluent to give a colorless solid. The product was dissolved in chloroform (3 ml) and heated for two minutes. The resulting solution was subjected to crystallization by slow evaporation of the solvent resulting in single crystals suitable for X-ray crystallographic studies.

### S3. Refinement

All H atoms bonded to C-atoms were positioned geometrically and refined using a riding model, with C—H = 0.93, 0.96, 0.97 and 0.98 Å, for aryl, methyl, methylene and methine H-atoms, respectively. The $U_{iso}$(H) were allowed at 1.5$U_{eq}$(C methyl) or 1.2$U_{eq}$(C non-methyl). The hydroxy H-atom was located from a difference map and was alloewed to refine freely. An absolute structure was not established due to insufficient anomalous dispersion effects. Therefore, 2699 Friedel pairs of reflections were merged.

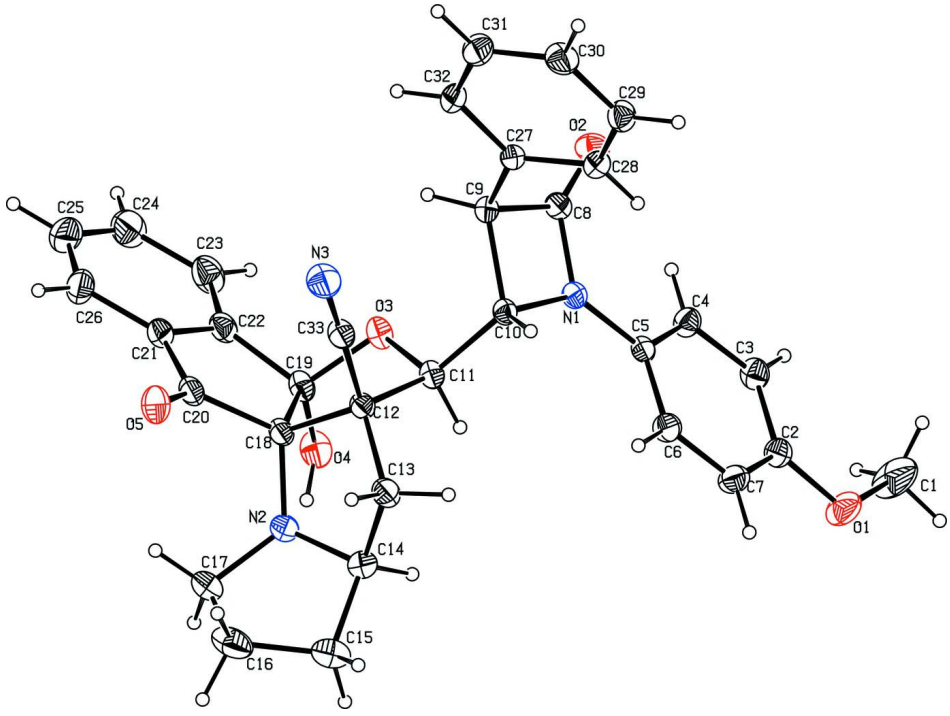


**Figure 1**

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level. H atoms are presented as a small spheres of arbitrary radius.

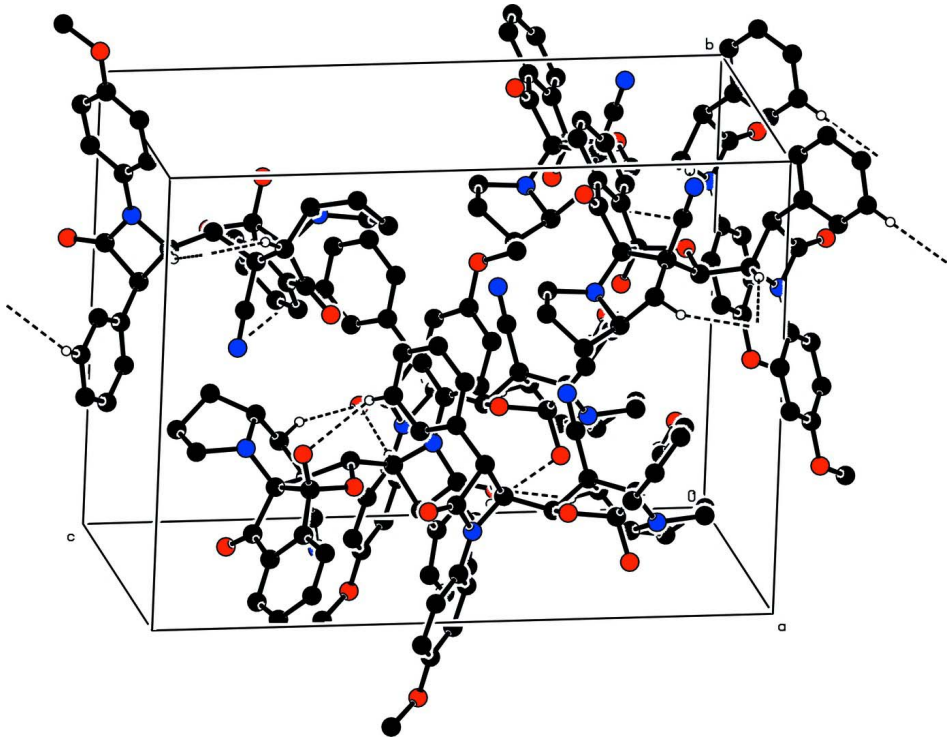


**Figure 2**

The crystal packing of the title compound viewed down *a* axis. H-atoms not involved in H-bonds have been excluded for clarity.

### 11-Hydroxy-9-[1-(4-methylphenyl)-4-oxo-3-phenylazetidin-2-yl]-18-oxo-10-oxa- 2-azapentacyclo[9.7.0.0$^{1,8}$.0$^{2,6}$.0$^{12,17}$]octadeca-12,14,16-triene-8- carbonitrile

*Crystal data*

$C_{33}H_{29}N_3O_5$
$M_r$ = 547.59
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a$ = 10.2874 (16) Å
$b$ = 14.138 (3) Å
$c$ = 18.866 (3) Å
$V$ = 2743.9 (8) Å$^3$
$Z$ = 4
$F(000)$ = 1152
$D_x$ = 1.326 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 6406 reflections
$\theta$ = 1.8–28.0°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 K
Block, colourless
0.30 × 0.25 × 0.20 mm

*Data collection*

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ and $\varphi$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.973, $T_{max}$ = 0.982
14810 measured reflections
6406 independent reflections
4551 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034
$\theta_{max}$ = 28.0°, $\theta_{min}$ = 1.8°
$h$ = −7→13
$k$ = −18→18
$l$ = −24→23

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.123$
$S = 1.00$
6406 reflections
375 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.2909P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{min} = -0.16$ e Å$^{-3}$

*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.

**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 $F^2 > 2$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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.5616 (4) | 0.7130 (2) | 0.3845 (2) | 0.1143 (16) |
| H1A | 0.6495 | 0.7115 | 0.4016 | 0.171* |
| H1B | 0.5356 | 0.7775 | 0.3770 | 0.171* |
| H1C | 0.5562 | 0.6789 | 0.3407 | 0.171* |
| C2 | 0.4988 (2) | 0.57555 (16) | 0.45053 (11) | 0.0485 (6) |
| C3 | 0.6029 (2) | 0.52399 (16) | 0.42370 (12) | 0.0491 (5) |
| H3 | 0.6624 | 0.5527 | 0.3935 | 0.059* |
| C4 | 0.6177 (2) | 0.42957 (16) | 0.44211 (12) | 0.0456 (5) |
| H4 | 0.6874 | 0.3951 | 0.4243 | 0.055* |
| C5 | 0.5287 (2) | 0.38631 (14) | 0.48714 (9) | 0.0364 (4) |
| C6 | 0.4241 (2) | 0.43760 (16) | 0.51336 (11) | 0.0461 (5) |
| H6 | 0.3636 | 0.4087 | 0.5429 | 0.055* |
| C7 | 0.4105 (3) | 0.53205 (17) | 0.49536 (13) | 0.0538 (6) |
| H7 | 0.3412 | 0.5667 | 0.5136 | 0.065* |
| C8 | 0.6046 (2) | 0.21567 (16) | 0.47055 (11) | 0.0394 (5) |
| C9 | 0.5463 (2) | 0.14150 (15) | 0.52086 (10) | 0.0386 (5) |
| H9 | 0.6120 | 0.1086 | 0.5493 | 0.046* |
| C10 | 0.4835 (2) | 0.22795 (14) | 0.56050 (10) | 0.0363 (4) |
| H10 | 0.3884 | 0.2277 | 0.5571 | 0.044* |
| C11 | 0.5303 (2) | 0.25097 (16) | 0.63567 (10) | 0.0394 (5) |
| H11 | 0.5225 | 0.3194 | 0.6429 | 0.047* |
| C12 | 0.4597 (2) | 0.19991 (15) | 0.69836 (10) | 0.0384 (5) |
| C13 | 0.3429 (2) | 0.25415 (18) | 0.72944 (12) | 0.0486 (6) |
| H13A | 0.2913 | 0.2827 | 0.6921 | 0.058* |

| | | | | |
|---|---|---|---|---|
| H13B | 0.2880 | 0.2125 | 0.7572 | 0.058* |
| C14 | 0.4057 (2) | 0.32998 (17) | 0.77630 (12) | 0.0510 (6) |
| H14 | 0.4282 | 0.3856 | 0.7478 | 0.061* |
| C15 | 0.3287 (3) | 0.3600 (2) | 0.84225 (15) | 0.0708 (8) |
| H15A | 0.3489 | 0.4247 | 0.8553 | 0.085* |
| H15B | 0.2359 | 0.3547 | 0.8340 | 0.085* |
| C16 | 0.3722 (4) | 0.2922 (2) | 0.89897 (15) | 0.0761 (9) |
| H16A | 0.3232 | 0.2336 | 0.8967 | 0.091* |
| H16B | 0.3619 | 0.3197 | 0.9457 | 0.091* |
| C17 | 0.5135 (4) | 0.2754 (2) | 0.88228 (13) | 0.0788 (10) |
| H17A | 0.5675 | 0.3220 | 0.9060 | 0.095* |
| H17B | 0.5398 | 0.2129 | 0.8978 | 0.095* |
| C18 | 0.5657 (2) | 0.20575 (15) | 0.75882 (10) | 0.0402 (5) |
| C19 | 0.6952 (2) | 0.22920 (17) | 0.71836 (11) | 0.0452 (5) |
| C20 | 0.5949 (2) | 0.11036 (17) | 0.79611 (10) | 0.0456 (5) |
| C21 | 0.7328 (2) | 0.08641 (17) | 0.78283 (11) | 0.0482 (6) |
| C22 | 0.7901 (2) | 0.15161 (18) | 0.73715 (12) | 0.0491 (6) |
| C23 | 0.9189 (3) | 0.1410 (2) | 0.71638 (14) | 0.0692 (8) |
| H23 | 0.9576 | 0.1838 | 0.6855 | 0.083* |
| C24 | 0.9880 (3) | 0.0645 (3) | 0.74306 (16) | 0.0816 (9) |
| H24 | 1.0744 | 0.0563 | 0.7299 | 0.098* |
| C25 | 0.9311 (3) | 0.0003 (3) | 0.78893 (17) | 0.0795 (9) |
| H25 | 0.9800 | −0.0500 | 0.8061 | 0.095* |
| C26 | 0.8033 (3) | 0.00942 (19) | 0.80953 (14) | 0.0639 (7) |
| H26 | 0.7651 | −0.0340 | 0.8401 | 0.077* |
| C27 | 0.4551 (2) | 0.07495 (14) | 0.48296 (10) | 0.0392 (5) |
| C28 | 0.3612 (2) | 0.10843 (16) | 0.43596 (11) | 0.0434 (5) |
| H28 | 0.3541 | 0.1732 | 0.4284 | 0.052* |
| C29 | 0.2783 (2) | 0.04784 (17) | 0.40026 (12) | 0.0515 (6) |
| H29 | 0.2163 | 0.0718 | 0.3692 | 0.062* |
| C30 | 0.2885 (3) | −0.04835 (18) | 0.41119 (14) | 0.0614 (7) |
| H30 | 0.2332 | −0.0895 | 0.3873 | 0.074* |
| C31 | 0.3803 (3) | −0.08373 (18) | 0.45731 (14) | 0.0678 (8) |
| H31 | 0.3862 | −0.1486 | 0.4648 | 0.081* |
| C32 | 0.4644 (3) | −0.02266 (16) | 0.49279 (12) | 0.0546 (6) |
| H32 | 0.5272 | −0.0471 | 0.5232 | 0.066* |
| C33 | 0.4248 (2) | 0.10128 (17) | 0.67921 (11) | 0.0453 (5) |
| N1 | 0.54457 (16) | 0.28914 (12) | 0.50512 (8) | 0.0385 (4) |
| N2 | 0.5264 (2) | 0.28416 (13) | 0.80428 (10) | 0.0507 (5) |
| N3 | 0.3949 (2) | 0.02553 (16) | 0.66654 (12) | 0.0653 (6) |
| O1 | 0.4773 (2) | 0.67002 (12) | 0.43580 (11) | 0.0735 (6) |
| O2 | 0.67202 (16) | 0.21280 (12) | 0.41786 (9) | 0.0542 (4) |
| O3 | 0.66478 (15) | 0.22531 (11) | 0.64393 (7) | 0.0463 (4) |
| O4 | 0.7423 (2) | 0.31898 (13) | 0.73731 (10) | 0.0601 (5) |
| O5 | 0.51536 (18) | 0.06443 (13) | 0.82903 (9) | 0.0626 (5) |
| H4A | 0.679 (3) | 0.335 (2) | 0.7702 (17) | 0.076 (10)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.151 (4) | 0.0565 (19) | 0.136 (3) | 0.018 (2) | 0.072 (3) | 0.040 (2) |
| C2 | 0.0609 (15) | 0.0398 (11) | 0.0449 (12) | −0.0010 (11) | 0.0029 (11) | 0.0046 (9) |
| C3 | 0.0504 (14) | 0.0484 (13) | 0.0483 (12) | −0.0070 (11) | 0.0073 (11) | 0.0095 (10) |
| C4 | 0.0416 (13) | 0.0462 (12) | 0.0490 (12) | 0.0032 (10) | 0.0024 (10) | 0.0061 (10) |
| C5 | 0.0401 (11) | 0.0389 (10) | 0.0302 (9) | −0.0009 (9) | −0.0017 (9) | 0.0016 (8) |
| C6 | 0.0495 (14) | 0.0436 (12) | 0.0453 (11) | −0.0018 (10) | 0.0096 (10) | 0.0053 (10) |
| C7 | 0.0576 (15) | 0.0469 (13) | 0.0569 (14) | 0.0086 (11) | 0.0116 (12) | 0.0016 (11) |
| C8 | 0.0322 (10) | 0.0483 (12) | 0.0378 (10) | 0.0010 (10) | 0.0003 (9) | −0.0014 (9) |
| C9 | 0.0412 (11) | 0.0396 (11) | 0.0351 (10) | 0.0050 (9) | −0.0026 (9) | 0.0026 (8) |
| C10 | 0.0394 (11) | 0.0368 (10) | 0.0327 (9) | −0.0013 (9) | 0.0003 (8) | 0.0035 (8) |
| C11 | 0.0420 (12) | 0.0413 (11) | 0.0348 (10) | −0.0033 (9) | −0.0035 (9) | 0.0030 (8) |
| C12 | 0.0450 (12) | 0.0366 (11) | 0.0336 (9) | −0.0028 (9) | −0.0017 (9) | −0.0003 (8) |
| C13 | 0.0480 (14) | 0.0573 (14) | 0.0406 (11) | 0.0020 (11) | 0.0004 (10) | −0.0001 (10) |
| C14 | 0.0569 (15) | 0.0469 (13) | 0.0491 (12) | 0.0064 (11) | −0.0008 (11) | −0.0037 (10) |
| C15 | 0.0767 (19) | 0.0723 (18) | 0.0634 (16) | 0.0013 (16) | 0.0074 (14) | −0.0204 (15) |
| C16 | 0.114 (3) | 0.0599 (17) | 0.0547 (15) | −0.0283 (17) | 0.0223 (16) | −0.0133 (13) |
| C17 | 0.121 (3) | 0.075 (2) | 0.0403 (13) | 0.0251 (19) | −0.0113 (16) | −0.0136 (13) |
| C18 | 0.0483 (13) | 0.0414 (11) | 0.0311 (9) | −0.0035 (10) | −0.0034 (9) | 0.0005 (9) |
| C19 | 0.0500 (13) | 0.0498 (13) | 0.0357 (10) | −0.0089 (11) | −0.0074 (10) | 0.0028 (9) |
| C20 | 0.0567 (15) | 0.0481 (13) | 0.0319 (10) | −0.0025 (11) | −0.0041 (10) | 0.0023 (9) |
| C21 | 0.0567 (15) | 0.0495 (13) | 0.0386 (11) | 0.0023 (11) | −0.0093 (10) | −0.0012 (10) |
| C22 | 0.0466 (14) | 0.0635 (15) | 0.0371 (10) | 0.0009 (11) | −0.0088 (10) | −0.0039 (10) |
| C23 | 0.0532 (17) | 0.101 (2) | 0.0531 (14) | 0.0014 (16) | −0.0042 (13) | 0.0038 (15) |
| C24 | 0.0574 (18) | 0.121 (3) | 0.0660 (17) | 0.0263 (19) | −0.0088 (15) | −0.0056 (19) |
| C25 | 0.076 (2) | 0.084 (2) | 0.0776 (19) | 0.0295 (18) | −0.0236 (17) | −0.0081 (17) |
| C26 | 0.077 (2) | 0.0542 (15) | 0.0610 (15) | 0.0083 (14) | −0.0172 (15) | 0.0064 (13) |
| C27 | 0.0474 (12) | 0.0361 (10) | 0.0342 (10) | 0.0048 (9) | 0.0024 (9) | 0.0014 (8) |
| C28 | 0.0452 (13) | 0.0392 (11) | 0.0459 (11) | 0.0036 (10) | −0.0009 (10) | 0.0013 (10) |
| C29 | 0.0522 (14) | 0.0528 (14) | 0.0494 (13) | 0.0003 (11) | −0.0089 (11) | 0.0015 (11) |
| C30 | 0.0813 (19) | 0.0471 (14) | 0.0558 (14) | −0.0145 (13) | −0.0054 (14) | −0.0070 (11) |
| C31 | 0.109 (2) | 0.0323 (12) | 0.0621 (15) | −0.0002 (14) | −0.0115 (16) | 0.0003 (11) |
| C32 | 0.0758 (17) | 0.0388 (12) | 0.0491 (12) | 0.0119 (12) | −0.0102 (12) | 0.0030 (10) |
| C33 | 0.0511 (14) | 0.0466 (13) | 0.0382 (10) | −0.0049 (10) | −0.0031 (10) | 0.0017 (10) |
| N1 | 0.0413 (10) | 0.0396 (9) | 0.0345 (8) | 0.0001 (8) | 0.0041 (7) | 0.0040 (7) |
| N2 | 0.0653 (13) | 0.0451 (10) | 0.0416 (9) | 0.0050 (10) | −0.0082 (9) | −0.0075 (8) |
| N3 | 0.0829 (17) | 0.0497 (13) | 0.0633 (13) | −0.0155 (12) | −0.0078 (12) | −0.0038 (11) |
| O1 | 0.0946 (15) | 0.0422 (9) | 0.0838 (12) | 0.0107 (10) | 0.0267 (12) | 0.0173 (9) |
| O2 | 0.0499 (10) | 0.0567 (10) | 0.0560 (10) | −0.0011 (8) | 0.0167 (8) | −0.0038 (8) |
| O3 | 0.0413 (9) | 0.0605 (10) | 0.0371 (8) | −0.0055 (7) | −0.0031 (6) | 0.0063 (7) |
| O4 | 0.0670 (12) | 0.0543 (10) | 0.0590 (10) | −0.0237 (9) | −0.0130 (10) | 0.0045 (9) |
| O5 | 0.0707 (12) | 0.0609 (11) | 0.0563 (9) | −0.0074 (10) | 0.0027 (9) | 0.0211 (8) |

*Geometric parameters (Å, º)*

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| C1—O1 | 1.434 (4) | C15—H15B | 0.9700 |
| C1—H1A | 0.9600 | C16—C17 | 1.506 (5) |
| C1—H1B | 0.9600 | C16—H16A | 0.9700 |
| C1—H1C | 0.9600 | C16—H16B | 0.9700 |
| C2—O1 | 1.382 (3) | C17—N2 | 1.483 (3) |
| C2—C7 | 1.385 (3) | C17—H17A | 0.9700 |
| C2—C3 | 1.391 (3) | C17—H17B | 0.9700 |
| C3—C4 | 1.388 (3) | C18—N2 | 1.459 (3) |
| C3—H3 | 0.9300 | C18—C20 | 1.550 (3) |
| C4—C5 | 1.391 (3) | C18—C19 | 1.571 (3) |
| C4—H4 | 0.9300 | C19—O4 | 1.405 (3) |
| C5—C6 | 1.388 (3) | C19—O3 | 1.440 (2) |
| C5—N1 | 1.424 (3) | C19—C22 | 1.511 (3) |
| C6—C7 | 1.385 (3) | C20—O5 | 1.215 (3) |
| C6—H6 | 0.9300 | C20—C21 | 1.480 (4) |
| C7—H7 | 0.9300 | C21—C22 | 1.393 (3) |
| C8—O2 | 1.213 (2) | C21—C26 | 1.402 (3) |
| C8—N1 | 1.373 (3) | C22—C23 | 1.390 (4) |
| C8—C9 | 1.536 (3) | C23—C24 | 1.388 (4) |
| C9—C27 | 1.509 (3) | C23—H23 | 0.9300 |
| C9—C10 | 1.572 (3) | C24—C25 | 1.385 (5) |
| C9—H9 | 0.9800 | C24—H24 | 0.9300 |
| C10—N1 | 1.495 (2) | C25—C26 | 1.377 (4) |
| C10—C11 | 1.533 (3) | C25—H25 | 0.9300 |
| C10—H10 | 0.9800 | C26—H26 | 0.9300 |
| C11—O3 | 1.439 (3) | C27—C28 | 1.394 (3) |
| C11—C12 | 1.564 (3) | C27—C32 | 1.396 (3) |
| C11—H11 | 0.9800 | C28—C29 | 1.384 (3) |
| C12—C33 | 1.484 (3) | C28—H28 | 0.9300 |
| C12—C13 | 1.541 (3) | C29—C30 | 1.380 (3) |
| C12—C18 | 1.581 (3) | C29—H29 | 0.9300 |
| C13—C14 | 1.532 (3) | C30—C31 | 1.378 (4) |
| C13—H13A | 0.9700 | C30—H30 | 0.9300 |
| C13—H13B | 0.9700 | C31—C32 | 1.394 (4) |
| C14—N2 | 1.497 (3) | C31—H31 | 0.9300 |
| C14—C15 | 1.535 (4) | C32—H32 | 0.9300 |
| C14—H14 | 0.9800 | C33—N3 | 1.140 (3) |
| C15—C16 | 1.505 (4) | O4—H4A | 0.93 (3) |
| C15—H15A | 0.9700 | | |

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| O1—C1—H1A | 109.5 | C17—C16—H16A | 111.0 |
| O1—C1—H1B | 109.5 | C15—C16—H16B | 111.0 |
| H1A—C1—H1B | 109.5 | C17—C16—H16B | 111.0 |
| O1—C1—H1C | 109.5 | H16A—C16—H16B | 109.0 |
| H1A—C1—H1C | 109.5 | N2—C17—C16 | 106.3 (2) |
| H1B—C1—H1C | 109.5 | N2—C17—H17A | 110.5 |

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| O1—C2—C7 | 116.5 (2) | C16—C17—H17A | 110.5 |
| O1—C2—C3 | 123.8 (2) | N2—C17—H17B | 110.5 |
| C7—C2—C3 | 119.6 (2) | C16—C17—H17B | 110.5 |
| C4—C3—C2 | 119.8 (2) | H17A—C17—H17B | 108.7 |
| C4—C3—H3 | 120.1 | N2—C18—C20 | 116.61 (16) |
| C2—C3—H3 | 120.1 | N2—C18—C19 | 111.12 (18) |
| C3—C4—C5 | 120.2 (2) | C20—C18—C19 | 103.89 (18) |
| C3—C4—H4 | 119.9 | N2—C18—C12 | 105.82 (17) |
| C5—C4—H4 | 119.9 | C20—C18—C12 | 114.56 (17) |
| C6—C5—C4 | 119.87 (19) | C19—C18—C12 | 104.23 (15) |
| C6—C5—N1 | 120.51 (18) | O4—C19—O3 | 110.96 (17) |
| C4—C5—N1 | 119.61 (18) | O4—C19—C22 | 111.93 (19) |
| C7—C6—C5 | 119.7 (2) | O3—C19—C22 | 109.98 (19) |
| C7—C6—H6 | 120.2 | O4—C19—C18 | 111.07 (19) |
| C5—C6—H6 | 120.2 | O3—C19—C18 | 106.35 (17) |
| C6—C7—C2 | 120.7 (2) | C22—C19—C18 | 106.31 (18) |
| C6—C7—H7 | 119.6 | O5—C20—C21 | 127.6 (2) |
| C2—C7—H7 | 119.6 | O5—C20—C18 | 124.5 (2) |
| O2—C8—N1 | 132.2 (2) | C21—C20—C18 | 107.93 (19) |
| O2—C8—C9 | 135.0 (2) | C22—C21—C26 | 121.1 (2) |
| N1—C8—C9 | 92.72 (15) | C22—C21—C20 | 111.0 (2) |
| C27—C9—C8 | 112.06 (16) | C26—C21—C20 | 127.8 (2) |
| C27—C9—C10 | 117.04 (18) | C23—C22—C21 | 120.4 (2) |
| C8—C9—C10 | 85.62 (15) | C23—C22—C19 | 128.9 (2) |
| C27—C9—H9 | 113.1 | C21—C22—C19 | 110.6 (2) |
| C8—C9—H9 | 113.1 | C24—C23—C22 | 118.1 (3) |
| C10—C9—H9 | 113.1 | C24—C23—H23 | 121.0 |
| N1—C10—C11 | 113.07 (16) | C22—C23—H23 | 121.0 |
| N1—C10—C9 | 86.83 (14) | C25—C24—C23 | 121.4 (3) |
| C11—C10—C9 | 118.45 (17) | C25—C24—H24 | 119.3 |
| N1—C10—H10 | 112.1 | C23—C24—H24 | 119.3 |
| C11—C10—H10 | 112.1 | C26—C25—C24 | 121.2 (3) |
| C9—C10—H10 | 112.1 | C26—C25—H25 | 119.4 |
| O3—C11—C10 | 110.40 (17) | C24—C25—H25 | 119.4 |
| O3—C11—C12 | 104.37 (16) | C25—C26—C21 | 117.7 (3) |
| C10—C11—C12 | 117.12 (17) | C25—C26—H26 | 121.1 |
| O3—C11—H11 | 108.2 | C21—C26—H26 | 121.1 |
| C10—C11—H11 | 108.2 | C28—C27—C32 | 117.9 (2) |
| C12—C11—H11 | 108.2 | C28—C27—C9 | 121.37 (18) |
| C33—C12—C13 | 111.82 (19) | C32—C27—C9 | 120.69 (19) |
| C33—C12—C11 | 111.20 (17) | C29—C28—C27 | 121.8 (2) |
| C13—C12—C11 | 114.83 (18) | C29—C28—H28 | 119.1 |
| C33—C12—C18 | 113.06 (18) | C27—C28—H28 | 119.1 |
| C13—C12—C18 | 103.73 (16) | C30—C29—C28 | 119.4 (2) |
| C11—C12—C18 | 101.59 (16) | C30—C29—H29 | 120.3 |
| C14—C13—C12 | 103.85 (19) | C28—C29—H29 | 120.3 |
| C14—C13—H13A | 111.0 | C31—C30—C29 | 120.3 (2) |
| C12—C13—H13A | 111.0 | C31—C30—H30 | 119.9 |

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| C14—C13—H13B | 111.0 | C29—C30—H30 | 119.9 |
| C12—C13—H13B | 111.0 | C30—C31—C32 | 120.3 (2) |
| H13A—C13—H13B | 109.0 | C30—C31—H31 | 119.9 |
| N2—C14—C13 | 104.49 (18) | C32—C31—H31 | 119.9 |
| N2—C14—C15 | 105.2 (2) | C31—C32—C27 | 120.4 (2) |
| C13—C14—C15 | 116.4 (2) | C31—C32—H32 | 119.8 |
| N2—C14—H14 | 110.1 | C27—C32—H32 | 119.8 |
| C13—C14—H14 | 110.1 | N3—C33—C12 | 177.5 (3) |
| C15—C14—H14 | 110.1 | C8—N1—C5 | 131.91 (16) |
| C16—C15—C14 | 104.3 (2) | C8—N1—C10 | 94.77 (16) |
| C16—C15—H15A | 110.9 | C5—N1—C10 | 132.56 (16) |
| C14—C15—H15A | 110.9 | C18—N2—C17 | 123.04 (19) |
| C16—C15—H15B | 110.9 | C18—N2—C14 | 110.58 (16) |
| C14—C15—H15B | 110.9 | C17—N2—C14 | 108.1 (2) |
| H15A—C15—H15B | 108.9 | C2—O1—C1 | 116.7 (2) |
| C15—C16—C17 | 103.8 (2) | C11—O3—C19 | 107.76 (16) |
| C15—C16—H16A | 111.0 | C19—O4—H4A | 98 (2) |

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| O1—C2—C3—C4 | 179.2 (2) | O5—C20—C21—C26 | −5.0 (4) |
| C7—C2—C3—C4 | −0.2 (4) | C18—C20—C21—C26 | 176.2 (2) |
| C2—C3—C4—C5 | 0.2 (4) | C26—C21—C22—C23 | 0.7 (4) |
| C3—C4—C5—C6 | 0.4 (3) | C20—C21—C22—C23 | −178.5 (2) |
| C3—C4—C5—N1 | 179.5 (2) | C26—C21—C22—C19 | −178.0 (2) |
| C4—C5—C6—C7 | −1.1 (3) | C20—C21—C22—C19 | 2.8 (3) |
| N1—C5—C6—C7 | 179.9 (2) | O4—C19—C22—C23 | −56.9 (3) |
| C5—C6—C7—C2 | 1.1 (4) | O3—C19—C22—C23 | 66.9 (3) |
| O1—C2—C7—C6 | −179.8 (2) | C18—C19—C22—C23 | −178.4 (2) |
| C3—C2—C7—C6 | −0.5 (4) | O4—C19—C22—C21 | 121.6 (2) |
| O2—C8—C9—C27 | −60.9 (3) | O3—C19—C22—C21 | −114.6 (2) |
| N1—C8—C9—C27 | 115.56 (18) | C18—C19—C22—C21 | 0.2 (2) |
| O2—C8—C9—C10 | −178.4 (3) | C21—C22—C23—C24 | −0.7 (4) |
| N1—C8—C9—C10 | −1.89 (15) | C19—C22—C23—C24 | 177.7 (2) |
| C27—C9—C10—N1 | −110.83 (17) | C22—C23—C24—C25 | 0.2 (4) |
| C8—C9—C10—N1 | 1.73 (14) | C23—C24—C25—C26 | 0.4 (5) |
| C27—C9—C10—C11 | 134.57 (19) | C24—C25—C26—C21 | −0.4 (4) |
| C8—C9—C10—C11 | −112.87 (18) | C22—C21—C26—C25 | −0.2 (4) |
| N1—C10—C11—O3 | −68.3 (2) | C20—C21—C26—C25 | 178.9 (2) |
| C9—C10—C11—O3 | 31.1 (2) | C8—C9—C27—C28 | −46.0 (3) |
| N1—C10—C11—C12 | 172.52 (17) | C10—C9—C27—C28 | 50.6 (3) |
| C9—C10—C11—C12 | −88.1 (2) | C8—C9—C27—C32 | 132.5 (2) |
| O3—C11—C12—C33 | −85.7 (2) | C10—C9—C27—C32 | −130.9 (2) |
| C10—C11—C12—C33 | 36.7 (3) | C32—C27—C28—C29 | 0.6 (3) |
| O3—C11—C12—C13 | 146.07 (18) | C9—C27—C28—C29 | 179.1 (2) |
| C10—C11—C12—C13 | −91.5 (2) | C27—C28—C29—C30 | −0.1 (4) |
| O3—C11—C12—C18 | 34.9 (2) | C28—C29—C30—C31 | 0.1 (4) |
| C10—C11—C12—C18 | 157.24 (18) | C29—C30—C31—C32 | −0.6 (4) |
| C33—C12—C13—C14 | 154.06 (18) | C30—C31—C32—C27 | 1.1 (4) |
| C11—C12—C13—C14 | −78.0 (2) | C28—C27—C32—C31 | −1.1 (4) |

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| C18—C12—C13—C14 | 31.9 (2) | C9—C27—C32—C31 | −179.6 (2) |
| C12—C13—C14—N2 | −33.1 (2) | O2—C8—N1—C5 | 7.9 (4) |
| C12—C13—C14—C15 | −148.5 (2) | C9—C8—N1—C5 | −168.7 (2) |
| N2—C14—C15—C16 | −26.4 (3) | O2—C8—N1—C10 | 178.6 (2) |
| C13—C14—C15—C16 | 88.7 (3) | C9—C8—N1—C10 | 1.98 (16) |
| C14—C15—C16—C17 | 35.1 (3) | C6—C5—N1—C8 | 153.4 (2) |
| C15—C16—C17—N2 | −31.0 (3) | C4—C5—N1—C8 | −25.6 (3) |
| C33—C12—C18—N2 | −140.51 (18) | C6—C5—N1—C10 | −14.0 (3) |
| C13—C12—C18—N2 | −19.2 (2) | C4—C5—N1—C10 | 167.0 (2) |
| C11—C12—C18—N2 | 100.25 (19) | C11—C10—N1—C8 | 117.73 (19) |
| C33—C12—C18—C20 | −10.6 (3) | C9—C10—N1—C8 | −1.94 (15) |
| C13—C12—C18—C20 | 110.7 (2) | C11—C10—N1—C5 | −71.6 (3) |
| C11—C12—C18—C20 | −129.86 (18) | C9—C10—N1—C5 | 168.7 (2) |
| C33—C12—C18—C19 | 102.2 (2) | C20—C18—N2—C17 | −0.3 (3) |
| C13—C12—C18—C19 | −136.46 (18) | C19—C18—N2—C17 | −119.0 (3) |
| C11—C12—C18—C19 | −17.0 (2) | C12—C18—N2—C17 | 128.4 (2) |
| N2—C18—C19—O4 | 1.3 (2) | C20—C18—N2—C14 | −130.1 (2) |
| C20—C18—C19—O4 | −124.83 (18) | C19—C18—N2—C14 | 111.1 (2) |
| C12—C18—C19—O4 | 114.88 (18) | C12—C18—N2—C14 | −1.5 (2) |
| N2—C18—C19—O3 | −119.50 (18) | C16—C17—N2—C18 | −116.3 (3) |
| C20—C18—C19—O3 | 114.33 (18) | C16—C17—N2—C14 | 14.6 (3) |
| C12—C18—C19—O3 | −6.0 (2) | C13—C14—N2—C18 | 21.7 (2) |
| N2—C18—C19—C22 | 123.32 (19) | C15—C14—N2—C18 | 144.8 (2) |
| C20—C18—C19—C22 | −2.8 (2) | C13—C14—N2—C17 | −115.7 (2) |
| C12—C18—C19—C22 | −123.13 (17) | C15—C14—N2—C17 | 7.4 (3) |
| N2—C18—C20—O5 | 63.0 (3) | C7—C2—O1—C1 | −174.6 (3) |
| C19—C18—C20—O5 | −174.4 (2) | C3—C2—O1—C1 | 6.1 (4) |
| C12—C18—C20—O5 | −61.4 (3) | C10—C11—O3—C19 | −167.85 (16) |
| N2—C18—C20—C21 | −118.1 (2) | C12—C11—O3—C19 | −41.2 (2) |
| C19—C18—C20—C21 | 4.5 (2) | O4—C19—O3—C11 | −91.4 (2) |
| C12—C18—C20—C21 | 117.51 (19) | C22—C19—O3—C11 | 144.18 (19) |
| O5—C20—C21—C22 | 174.1 (2) | C18—C19—O3—C11 | 29.5 (2) |
| C18—C20—C21—C22 | −4.7 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H10···O2$^{i}$ | 0.98 | 2.43 | 3.337 (3) | 154 |
| C13—H13*A*···O2$^{i}$ | 0.97 | 2.41 | 3.321 (3) | 156 |
| C29—H29···O4$^{i}$ | 0.93 | 2.55 | 3.228 (3) | 130 |
| O4—H4*A*···N2 | 0.93 (3) | 1.84 (3) | 2.602 (3) | 137 (3) |

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