# data reports

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# Crystal structure of sepaconitine, a C$_{19}$-diterpenoid alkaloid from the roots of *Aconitum sinomontanum* Nakai

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The title compound [systematic name: [(1α,14α,16β)-20-ethyl-8,9,10-trihydroxy-1,14,16-trimethoxyaconitan-4-yl 2-amino-benzoate], C$_{30}$H$_{42}$N$_2$O$_8$, a natural C$_{19}$-diterpenoid alkaloid, possesses an aconitane carbon skeleton with four six-membered rings and two five-membered rings. The fused ring system contains two chair, one boat, one twist-boat and two envelope conformations. Intramolecular N—H···O hydrogen bonds are observed between the amino and carbonyl groups. The molecules are linked together *via* O—H···O hydrogen bonds, forming a three-dimensional framework.

## 1. Related literature

For the synthesis of the title compound, see: Wei *et al.* (1996). The absolute configuration of the title compound has been assigned to be the same as that reported for typical natural C$_{19}$-diterpenoid alkaloids, see: Wang *et al.* (2007); He *et al.* (2008). The six-ring rigid-frame structure of the title compound is identical to that of lappaconitine and mesaconitine (Wang *et al.*, 2007; He *et al.*, 2008).
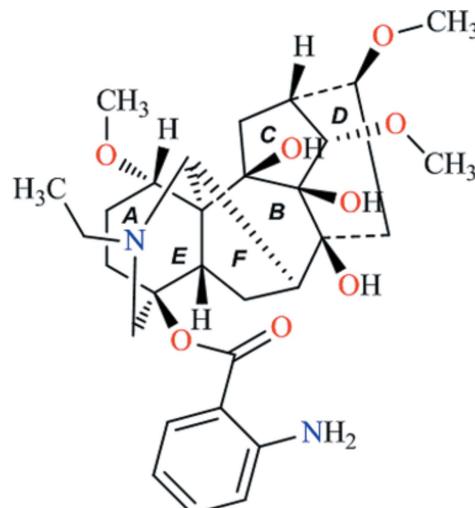


## 2. Experimental

### 2.1. Crystal data

C$_{30}$H$_{42}$N$_2$O$_8$
$M_r$ = 558.66
Orthorhombic, $P2_12_12_1$
$a$ = 9.6917 (5) Å
$b$ = 16.0510 (7) Å
$c$ = 18.3549 (7) Å

$V$ = 2855.3 (2) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.77 mm$^{-1}$
$T$ = 173 K
0.32 × 0.32 × 0.28 mm

### 2.2. Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min}$ = 0.791, $T_{max}$ = 0.813

8125 measured reflections
4528 independent reflections
3831 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.126
$S$ = 1.05
4528 reflections
388 parameters

60 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O5—H5A···O6 | 0.84 | 2.34 | 2.909 (3) | 126 |
| O4—H4···O5 | 0.84 | 2.27 | 2.684 (3) | 111 |
| O4—H4···O3$^i$ | 0.84 | 1.94 | 2.713 (3) | 153 |
| O3—H3···O4 | 0.84 | 1.99 | 2.524 (3) | 121 |
| N1—H1B···O1 | 0.88 | 2.00 | 2.666 (4) | 131 |
| N1—H1A···O8$^{ii}$ | 0.88 | 2.19 | 2.999 (3) | 152 |

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + 2$; (ii) $-x + \frac{3}{2}, -y, z + \frac{1}{2}$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

## Acknowledgements

## References

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He, D.-H., Zhu, Y.-C. & Hu, A.-X. (2008). *Acta Cryst.* E**64**, o1033–o1034.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Wang, Y.-P., Sun, W.-X., Zhang, J., Liu, H.-S. & Wen, H.-H. (2007). *Acta Cryst.* E**63**, o1645–o1647.

Wei, X.-Y., Wei, B.-Y. & Zhang, J. (1996). *Acta Botanica Sin.* **38**, 995–997.

# supporting information

# Crystal structure of sepaconitine, a C₁₉-diterpenoid alkaloid from the roots of *Aconitum sinomontanum* Nakai

## Xin-Wei Shi, Qiang-Qiang Lu, Jun-Hui Zhou and Xin-Ai Cui

## S1. Comment

The title compound was isolated from the roots of *Aconitum sinomontanum* Nakai, collected in Taibai mountain of the Qinling area, Shaanxi province, People's Republic of China. the crystal structure determination of sepaconitine was carried out and the result reported here.

The molecular structure is shown in Fig. 1. The molecule has a rigid structure consisting of six main rings (*A—F*), which is identical with that of lappaconitine and mesaconitine (Wang *et al.*, 2007; He *et al.*, 2008). The six-membered rings *A* (C1/C2/C3/C4/C5/C11) and N-containing heterocyclic ring *E* (C4/C5/C11/C17/N2/C18) adopt chair conformations; The six-membered ring *D* (C8/C9/C14/C13/C16/C15) displays a boat conformation, but *B* (C7/C8/C9/C10/C11/C17) adopts a twist-boat conformation; the five-membered rings *C* (C9/C10/C12/C13/C14) and *F* (C5/C6/C7/C17/C11) adopt C14- and C 17-envelope conformations, respectively. Two *cis*-fused ring junctions involve rings *A*/*E* and also *B*/*C*. Two *trans*-fused ring junctions are observed between rings *A*/*B* and between *E*/*F*. Ring *E* is slightly flattened at C5 due to the presence of an ethyl-substituted N atom in the ring. The benzoate moiety attached to C4 is almost planar. The OCH₃ group attached to C16 is disordered into two positions with site occupancies Factor of 0.5.

The crystal structure has an intra-molecular N—H···O hydrogen bond between the amino group and carbonyl O atom (Table 1). Inter-molecular N—H···O hydrogen bonds are observed in the crystal. The molecules are linked together via O —H``O hydrogen bonds in the *c* direction (Table 1, Fig. 2).

## S2. Experimental

The title compound was isolated from the roots of *Aconitum sinomontanum*, using a method described previously (Wei *et al.*, 1996). Colourless crystals were grown from methanol at room temperature by slow evaporation.

## S3. Refinement

The hydrogen atoms were placed in calculated positions and refined as riding with U$_{iso}$(H) = 1.2 Ueq (C) or 1.5Ueq(C, O). The positions of methyl and hydroxy hydrogens were rotationally optimized. The absolute configuration of the title compound, sepaconitine, has been assigned to be the same as that reported for typical natural C₁₉-diterpenoid alkaloids (Wang *et al.*, 2007; He *et al.*, 2008).
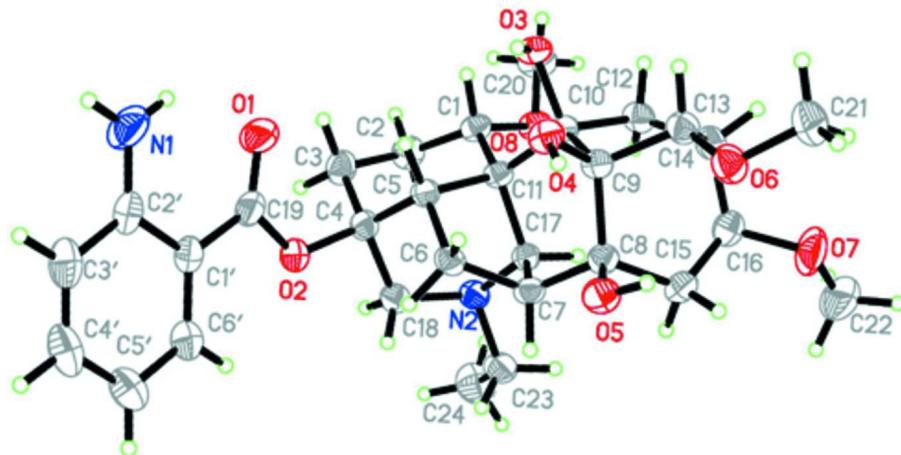
**Figure 1**

*ORTEPII* drawing of sepaconitine (I) with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.
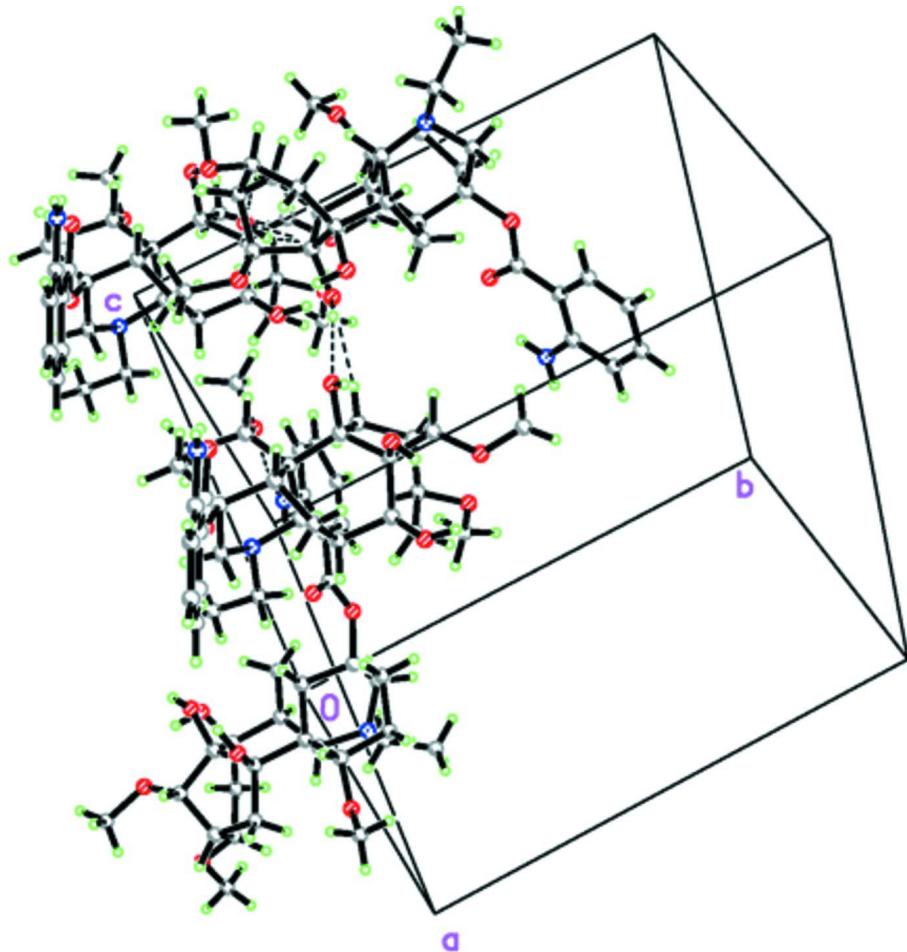
**Figure 2**

The packing of molecules in the crystal structure of sepaconitine (I), viewed along the *c* direction (Hydrogen bonds are shown as dashed lines).

## (1*α*,14*α*,16*β*)-20-Ethyl-8,9,10-trihydroxy-1,14,16-trimethoxyaconitan-4-yl 2-aminobenzoate

*Crystal data*

| | |
|---|---|
| $C_{30}H_{42}N_2O_8$ | $D_x = 1.300$ Mg m$^{-3}$ |
| $M_r = 558.66$ | Melting point = 250–252 K |
| Orthorhombic, $P2_12_12_1$ | Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å |
| Hall symbol: P 2ac 2ab | Cell parameters from 2628 reflections |
| $a = 9.6917$ (5) Å | $\theta = 3.7$–64.2° |
| $b = 16.0510$ (7) Å | $\mu = 0.77$ mm$^{-1}$ |
| $c = 18.3549$ (7) Å | $T = 173$ K |
| $V = 2855.3$ (2) Å$^3$ | Block, colorless |
| $Z = 4$ | 0.32 × 0.32 × 0.28 mm |
| $F(000) = 1200$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 8125 measured reflections |
| Radiation source: fine-focus sealed tube | 4528 independent reflections |
| Graphite monochromator | 3831 reflections with $I > 2\sigma(I)$ |
| phi and $\omega$ scans | $R_{int} = 0.029$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2002) | $\theta_{max} = 65.0°$, $\theta_{min} = 3.7°$ |
| | $h = -7\rightarrow11$ |
| $T_{min} = 0.791$, $T_{max} = 0.813$ | $k = -18\rightarrow18$ |
| | $l = -21\rightarrow18$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.046$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.126$ | |
| $S = 1.05$ | H-atom parameters constrained |
| 4528 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0752P)^2 + 0.0411P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 388 parameters | |
| 60 restraints | $(\Delta/\sigma)_{max} = 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.46$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.19$ e Å$^{-3}$ |

*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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.5519 (3) | 0.04441 (18) | 0.93627 (14) | 0.0459 (6) | |
| H1 | 0.5224 | 0.0713 | 0.9829 | 0.055* | |
| C2 | 0.5401 (3) | −0.04952 (19) | 0.94729 (17) | 0.0574 (7) | |
| H2A | 0.4494 | −0.0626 | 0.9693 | 0.069* | |

| H2B | 0.5451 | −0.0777 | 0.8994 | 0.069* | |
| C3 | 0.6545 (3) | −0.08242 (19) | 0.99638 (17) | 0.0587 (7) | |
| H3A | 0.6440 | −0.0598 | 1.0462 | 0.070* | |
| H3B | 0.6503 | −0.1440 | 0.9990 | 0.070* | |
| C4 | 0.7912 (3) | −0.05507 (17) | 0.96421 (14) | 0.0472 (6) | |
| C5 | 0.8106 (3) | 0.03991 (16) | 0.96718 (13) | 0.0417 (6) | |
| H5 | 0.8054 | 0.0615 | 1.0182 | 0.050* | |
| C6 | 0.9497 (3) | 0.06134 (17) | 0.93102 (14) | 0.0457 (6) | |
| H6A | 1.0101 | 0.0117 | 0.9287 | 0.055* | |
| H6B | 0.9979 | 0.1059 | 0.9583 | 0.055* | |
| C7 | 0.9109 (3) | 0.09122 (16) | 0.85379 (13) | 0.0443 (6) | |
| H7 | 0.9740 | 0.0659 | 0.8168 | 0.053* | |
| C8 | 0.9157 (3) | 0.18622 (16) | 0.84910 (14) | 0.0445 (6) | |
| C9 | 0.8267 (3) | 0.22508 (16) | 0.91195 (13) | 0.0416 (6) | |
| C10 | 0.6911 (3) | 0.17703 (16) | 0.92456 (12) | 0.0405 (6) | |
| C11 | 0.6968 (3) | 0.07934 (16) | 0.91761 (13) | 0.0399 (5) | |
| C12 | 0.5904 (3) | 0.22027 (18) | 0.87023 (15) | 0.0496 (6) | |
| H12A | 0.5568 | 0.1796 | 0.8338 | 0.060* | |
| H12B | 0.5098 | 0.2431 | 0.8967 | 0.060* | |
| C13 | 0.6700 (3) | 0.29100 (18) | 0.83222 (15) | 0.0527 (7) | |
| H13 | 0.6084 | 0.3399 | 0.8230 | 0.063* | |
| C14 | 0.7783 (3) | 0.31184 (17) | 0.88965 (15) | 0.0512 (7) | |
| H14 | 0.7319 | 0.3389 | 0.9322 | 0.061* | |
| C15 | 0.8737 (3) | 0.2179 (2) | 0.77238 (14) | 0.0565 (7) | |
| H15A | 0.9459 | 0.2574 | 0.7560 | 0.068* | |
| H15B | 0.8767 | 0.1696 | 0.7389 | 0.068* | |
| C16 | 0.7352 (4) | 0.2605 (2) | 0.76183 (15) | 0.0604 (8) | |
| H16 | 0.6702 | 0.2225 | 0.7356 | 0.073* | |
| C17 | 0.7620 (3) | 0.05823 (16) | 0.84348 (12) | 0.0415 (6) | |
| H17 | 0.7146 | 0.0900 | 0.8039 | 0.050* | |
| C18 | 0.8127 (3) | −0.08526 (17) | 0.88536 (14) | 0.0515 (7) | |
| H18A | 0.9130 | −0.0893 | 0.8758 | 0.062* | |
| H18B | 0.7734 | −0.1419 | 0.8806 | 0.062* | |
| C19 | 0.9371 (3) | −0.08098 (19) | 1.07202 (15) | 0.0556 (7) | |
| C20 | 0.3182 (3) | 0.0800 (3) | 0.9099 (2) | 0.0738 (10) | |
| H20A | 0.2839 | 0.0276 | 0.9305 | 0.111* | |
| H20B | 0.2567 | 0.0982 | 0.8706 | 0.111* | |
| H20C | 0.3209 | 0.1227 | 0.9480 | 0.111* | |
| C21 | 0.8619 (4) | 0.4468 (2) | 0.8583 (2) | 0.0807 (10) | |
| H21A | 0.8080 | 0.4534 | 0.8135 | 0.121* | |
| H21B | 0.9477 | 0.4788 | 0.8543 | 0.121* | |
| H21C | 0.8081 | 0.4672 | 0.8999 | 0.121* | |
| C22 | 0.7499 (12) | 0.3113 (7) | 0.6432 (4) | 0.114 (3) | 0.578 (12) |
| H22A | 0.8162 | 0.2697 | 0.6260 | 0.170* | 0.578 (12) |
| H22B | 0.7575 | 0.3617 | 0.6134 | 0.170* | 0.578 (12) |
| H22C | 0.6561 | 0.2888 | 0.6393 | 0.170* | 0.578 (12) |
| C22′ | 0.6743 (13) | 0.3573 (9) | 0.6707 (7) | 0.104 (4) | 0.422 (12) |
| H22D | 0.6386 | 0.3124 | 0.6398 | 0.155* | 0.422 (12) |

| | | | | | |
|---|---|---|---|---|---|
| H22E | 0.7364 | 0.3927 | 0.6421 | 0.155* | 0.422 (12) |
| H22F | 0.5972 | 0.3909 | 0.6890 | 0.155* | 0.422 (12) |
| C23 | 0.7964 (4) | −0.0559 (2) | 0.75690 (15) | 0.0625 (8) | |
| H23A | 0.8964 | −0.0683 | 0.7583 | 0.075* | |
| H23B | 0.7822 | −0.0086 | 0.7230 | 0.075* | |
| C24 | 0.7206 (6) | −0.1305 (2) | 0.7287 (2) | 0.0994 (14) | |
| H24A | 0.7516 | −0.1803 | 0.7548 | 0.149* | |
| H24B | 0.7394 | −0.1371 | 0.6765 | 0.149* | |
| H24C | 0.6213 | −0.1229 | 0.7362 | 0.149* | |
| C1′ | 1.0692 (3) | −0.11619 (17) | 1.09396 (15) | 0.0525 (7) | |
| C2′ | 1.1153 (4) | −0.10849 (19) | 1.16724 (16) | 0.0600 (8) | |
| C3′ | 1.2446 (4) | −0.1426 (2) | 1.1845 (2) | 0.0732 (10) | |
| H3′ | 1.2769 | −0.1389 | 1.2333 | 0.088* | |
| C4′ | 1.3246 (4) | −0.1804 (2) | 1.1341 (2) | 0.0786 (11) | |
| H4′ | 1.4116 | −0.2025 | 1.1480 | 0.094* | |
| C5′ | 1.2809 (4) | −0.1874 (2) | 1.0619 (2) | 0.0739 (9) | |
| H5′ | 1.3379 | −0.2134 | 1.0265 | 0.089* | |
| C6′ | 1.1544 (4) | −0.15597 (19) | 1.04293 (17) | 0.0624 (8) | |
| H6′ | 1.1236 | −0.1613 | 0.9940 | 0.075* | |
| N1 | 1.0397 (4) | −0.0685 (2) | 1.21841 (14) | 0.0858 (10) | |
| H1A | 1.0716 | −0.0636 | 1.2631 | 0.103* | |
| H1B | 0.9588 | −0.0473 | 1.2070 | 0.103* | |
| N2 | 0.7503 (2) | −0.03157 (13) | 0.82988 (10) | 0.0461 (5) | |
| O1 | 0.8590 (3) | −0.04165 (18) | 1.11148 (11) | 0.0792 (7) | |
| O2 | 0.9070 (2) | −0.09711 (12) | 1.00172 (10) | 0.0533 (5) | |
| O3 | 0.64582 (19) | 0.19706 (12) | 0.99729 (9) | 0.0492 (4) | |
| H3 | 0.7146 | 0.2082 | 1.0234 | 0.074* | |
| O4 | 0.8988 (2) | 0.22811 (12) | 0.97916 (9) | 0.0493 (5) | |
| H4 | 0.9790 | 0.2463 | 0.9721 | 0.074* | |
| O5 | 1.05780 (19) | 0.20907 (13) | 0.86056 (11) | 0.0567 (5) | |
| H5A | 1.0672 | 0.2605 | 0.8540 | 0.085* | |
| O6 | 0.8938 (2) | 0.36055 (12) | 0.86885 (12) | 0.0613 (5) | |
| O7 | 0.7770 (13) | 0.3303 (12) | 0.7134 (9) | 0.092 (3) | 0.578 (12) |
| O7′ | 0.7307 (16) | 0.3306 (16) | 0.7166 (13) | 0.084 (3) | 0.422 (12) |
| O8 | 0.45273 (18) | 0.06771 (13) | 0.88177 (10) | 0.0525 (5) | |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0340 (14) | 0.0631 (16) | 0.0404 (12) | −0.0009 (13) | 0.0003 (11) | −0.0027 (11) |
| C2 | 0.0407 (16) | 0.0666 (18) | 0.0650 (17) | −0.0101 (15) | 0.0019 (13) | 0.0052 (14) |
| C3 | 0.0552 (19) | 0.0600 (16) | 0.0609 (17) | −0.0055 (15) | 0.0027 (15) | 0.0118 (13) |
| C4 | 0.0425 (15) | 0.0532 (15) | 0.0457 (14) | 0.0050 (13) | −0.0030 (11) | 0.0054 (11) |
| C5 | 0.0368 (14) | 0.0518 (14) | 0.0365 (12) | 0.0057 (12) | −0.0025 (10) | 0.0001 (10) |
| C6 | 0.0378 (14) | 0.0488 (14) | 0.0506 (14) | 0.0084 (12) | −0.0013 (12) | 0.0003 (11) |
| C7 | 0.0367 (14) | 0.0537 (14) | 0.0424 (13) | 0.0037 (12) | 0.0040 (11) | −0.0013 (11) |
| C8 | 0.0348 (14) | 0.0524 (14) | 0.0462 (13) | −0.0005 (12) | 0.0019 (11) | 0.0017 (11) |
| C9 | 0.0351 (13) | 0.0507 (13) | 0.0390 (12) | 0.0015 (12) | −0.0043 (10) | −0.0035 (10) |

| | | | | | | |
|------|-------------|-------------|-------------|--------------|--------------|--------------|
| C10 | 0.0356 (14) | 0.0515 (14) | 0.0344 (12) | 0.0059 (11) | −0.0004 (10) | −0.0047 (10) |
| C11 | 0.0297 (13) | 0.0502 (13) | 0.0399 (12) | −0.0002 (11) | 0.0002 (10) | −0.0014 (10) |
| C12 | 0.0364 (14) | 0.0590 (16) | 0.0535 (15) | 0.0064 (13) | −0.0080 (12) | −0.0011 (12) |
| C13 | 0.0502 (17) | 0.0558 (15) | 0.0522 (15) | 0.0080 (14) | −0.0063 (13) | 0.0050 (11) |
| C14 | 0.0480 (16) | 0.0524 (15) | 0.0532 (15) | 0.0039 (13) | −0.0008 (13) | −0.0009 (12) |
| C15 | 0.0641 (19) | 0.0610 (16) | 0.0442 (14) | −0.0007 (15) | 0.0094 (13) | 0.0038 (12) |
| C16 | 0.069 (2) | 0.0665 (18) | 0.0457 (14) | 0.0031 (16) | −0.0044 (14) | 0.0074 (13) |
| C17 | 0.0373 (14) | 0.0496 (14) | 0.0376 (12) | 0.0042 (12) | −0.0020 (10) | 0.0008 (10) |
| C18 | 0.0553 (17) | 0.0493 (14) | 0.0499 (14) | 0.0053 (13) | 0.0003 (13) | −0.0020 (11) |
| C19 | 0.0615 (19) | 0.0633 (17) | 0.0420 (14) | 0.0028 (16) | −0.0019 (14) | 0.0099 (12) |
| C20 | 0.0339 (16) | 0.106 (3) | 0.082 (2) | 0.0081 (18) | 0.0051 (15) | −0.0039 (19) |
| C21 | 0.101 (3) | 0.0491 (16) | 0.092 (2) | 0.0004 (19) | 0.003 (2) | 0.0063 (16) |
| C22 | 0.135 (6) | 0.138 (6) | 0.067 (4) | 0.031 (5) | 0.013 (4) | 0.034 (4) |
| C22′ | 0.097 (6) | 0.126 (6) | 0.088 (6) | 0.041 (5) | 0.003 (5) | 0.037 (5) |
| C23 | 0.072 (2) | 0.0711 (19) | 0.0441 (14) | 0.0178 (17) | −0.0071 (14) | −0.0102 (13) |
| C24 | 0.149 (4) | 0.077 (2) | 0.072 (2) | 0.017 (3) | −0.021 (3) | −0.0308 (19) |
| C1′ | 0.0545 (17) | 0.0534 (15) | 0.0496 (14) | −0.0008 (14) | −0.0054 (13) | 0.0126 (12) |
| C2′ | 0.067 (2) | 0.0612 (17) | 0.0519 (16) | −0.0053 (16) | −0.0100 (15) | 0.0187 (13) |
| C3′ | 0.080 (3) | 0.069 (2) | 0.071 (2) | −0.004 (2) | −0.031 (2) | 0.0131 (17) |
| C4′ | 0.069 (2) | 0.0639 (19) | 0.103 (3) | 0.0035 (19) | −0.032 (2) | 0.0146 (19) |
| C5′ | 0.061 (2) | 0.069 (2) | 0.091 (2) | 0.0112 (18) | −0.0127 (18) | 0.0022 (17) |
| C6′ | 0.064 (2) | 0.0612 (17) | 0.0618 (18) | 0.0054 (17) | −0.0093 (16) | 0.0075 (14) |
| N1 | 0.089 (2) | 0.125 (3) | 0.0431 (13) | 0.005 (2) | −0.0066 (14) | 0.0039 (15) |
| N2 | 0.0471 (13) | 0.0501 (12) | 0.0412 (11) | 0.0055 (11) | −0.0047 (10) | −0.0058 (8) |
| O1 | 0.0785 (16) | 0.1133 (18) | 0.0457 (11) | 0.0293 (15) | −0.0017 (11) | −0.0006 (11) |
| O2 | 0.0560 (12) | 0.0576 (11) | 0.0462 (10) | 0.0098 (9) | −0.0034 (9) | 0.0067 (8) |
| O3 | 0.0427 (11) | 0.0645 (11) | 0.0404 (9) | 0.0048 (9) | 0.0031 (8) | −0.0097 (8) |
| O4 | 0.0418 (11) | 0.0626 (11) | 0.0436 (9) | −0.0016 (9) | −0.0073 (8) | −0.0034 (8) |
| O5 | 0.0392 (11) | 0.0613 (11) | 0.0695 (12) | −0.0060 (9) | 0.0066 (9) | 0.0040 (10) |
| O6 | 0.0618 (13) | 0.0485 (10) | 0.0737 (13) | −0.0011 (10) | 0.0018 (10) | 0.0024 (9) |
| O7 | 0.104 (6) | 0.104 (3) | 0.069 (3) | 0.011 (6) | −0.007 (5) | 0.047 (3) |
| O7′ | 0.087 (7) | 0.096 (4) | 0.067 (4) | 0.013 (6) | 0.001 (6) | 0.036 (3) |
| O8 | 0.0320 (10) | 0.0767 (12) | 0.0488 (10) | 0.0033 (9) | −0.0025 (8) | −0.0018 (9) |

*Geometric parameters (Å, º)*

| | | | |
|-----------|-----------|-------------|--------|
| C1—O8 | 1.437 (3) | C17—H17 | 1.0000 |
| C1—C2 | 1.525 (4) | C18—N2 | 1.465 (3) |
| C1—C11 | 1.551 (3) | C18—H18A | 0.9900 |
| C1—H1 | 1.0000 | C18—H18B | 0.9900 |
| C2—C3 | 1.523 (4) | C19—O1 | 1.223 (4) |
| C2—H2A | 0.9900 | C19—O2 | 1.348 (3) |
| C2—H2B | 0.9900 | C19—C1′ | 1.456 (4) |
| C3—C4 | 1.515 (4) | C20—O8 | 1.416 (3) |
| C3—H3A | 0.9900 | C20—H20A | 0.9800 |
| C3—H3B | 0.9900 | C20—H20B | 0.9800 |
| C4—O2 | 1.479 (3) | C20—H20C | 0.9800 |
| C4—C5 | 1.537 (4) | C21—O6 | 1.431 (4) |

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|---|---|---|---|
| C4—C18 | 1.540 (4) | C21—H21A | 0.9800 |
| C5—C6 | 1.542 (4) | C21—H21B | 0.9800 |
| C5—C11 | 1.563 (3) | C21—H21C | 0.9800 |
| C5—H5 | 1.0000 | C22—O7 | 1.350 (19) |
| C6—C7 | 1.543 (3) | C22—H22A | 0.9800 |
| C6—H6A | 0.9900 | C22—H22B | 0.9800 |
| C6—H6B | 0.9900 | C22—H22C | 0.9800 |
| C7—C8 | 1.528 (4) | C22′—O7′ | 1.09 (2) |
| C7—C17 | 1.549 (4) | C22′—H22D | 0.9800 |
| C7—H7 | 1.0000 | C22′—H22E | 0.9800 |
| C8—O5 | 1.440 (3) | C22′—H22F | 0.9800 |
| C8—C15 | 1.552 (4) | C23—N2 | 1.465 (3) |
| C8—C9 | 1.570 (4) | C23—C24 | 1.497 (5) |
| C9—O4 | 1.419 (3) | C23—H23A | 0.9900 |
| C9—C14 | 1.525 (4) | C23—H23B | 0.9900 |
| C9—C10 | 1.542 (4) | C24—H24A | 0.9800 |
| C10—O3 | 1.442 (3) | C24—H24B | 0.9800 |
| C10—C12 | 1.558 (3) | C24—H24C | 0.9800 |
| C10—C11 | 1.574 (4) | C1′—C6′ | 1.402 (4) |
| C11—C17 | 1.538 (3) | C1′—C2′ | 1.423 (4) |
| C12—C13 | 1.540 (4) | C2′—N1 | 1.353 (4) |
| C12—H12A | 0.9900 | C2′—C3′ | 1.404 (5) |
| C12—H12B | 0.9900 | C3′—C4′ | 1.352 (5) |
| C13—C16 | 1.520 (4) | C3′—H3′ | 0.9500 |
| C13—C14 | 1.525 (4) | C4′—C5′ | 1.397 (5) |
| C13—H13 | 1.0000 | C4′—H4′ | 0.9500 |
| C14—O6 | 1.418 (4) | C5′—C6′ | 1.371 (5) |
| C14—H14 | 1.0000 | C5′—H5′ | 0.9500 |
| C15—C16 | 1.518 (5) | C6′—H6′ | 0.9500 |
| C15—H15A | 0.9900 | N1—H1A | 0.8800 |
| C15—H15B | 0.9900 | N1—H1B | 0.8800 |
| C16—O7′ | 1.40 (2) | O3—H3 | 0.8400 |
| C16—O7 | 1.487 (17) | O4—H4 | 0.8400 |
| C16—H16 | 1.0000 | O5—H5A | 0.8400 |
| C17—N2 | 1.467 (3) | | |
| | | | |
| O8—C1—C2 | 107.4 (2) | C8—C15—H15B | 107.4 |
| O8—C1—C11 | 111.0 (2) | H15A—C15—H15B | 106.9 |
| C2—C1—C11 | 117.0 (2) | O7′—C16—C15 | 117.7 (7) |
| O8—C1—H1 | 107.0 | O7—C16—C15 | 100.0 (5) |
| C2—C1—H1 | 107.0 | O7′—C16—C13 | 103.4 (9) |
| C11—C1—H1 | 107.0 | O7—C16—C13 | 112.2 (7) |
| C3—C2—C1 | 111.5 (2) | C15—C16—C13 | 113.8 (2) |
| C3—C2—H2A | 109.3 | O7′—C16—H16 | 100.7 |
| C1—C2—H2A | 109.3 | O7—C16—H16 | 110.1 |
| C3—C2—H2B | 109.3 | C15—C16—H16 | 110.1 |
| C1—C2—H2B | 109.3 | C13—C16—H16 | 110.1 |
| H2A—C2—H2B | 108.0 | N2—C17—C11 | 109.6 (2) |

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| C4—C3—C2 | 107.8 (2) | N2—C17—C7 | 115.4 (2) |
| C4—C3—H3A | 110.1 | C11—C17—C7 | 101.48 (18) |
| C2—C3—H3A | 110.1 | N2—C17—H17 | 110.0 |
| C4—C3—H3B | 110.1 | C11—C17—H17 | 110.0 |
| C2—C3—H3B | 110.1 | C7—C17—H17 | 110.0 |
| H3A—C3—H3B | 108.5 | N2—C18—C4 | 114.3 (2) |
| O2—C4—C3 | 110.5 (2) | N2—C18—H18A | 108.7 |
| O2—C4—C5 | 110.1 (2) | C4—C18—H18A | 108.7 |
| C3—C4—C5 | 112.4 (2) | N2—C18—H18B | 108.7 |
| O2—C4—C18 | 101.0 (2) | C4—C18—H18B | 108.7 |
| C3—C4—C18 | 113.2 (2) | H18A—C18—H18B | 107.6 |
| C5—C4—C18 | 109.2 (2) | O1—C19—O2 | 122.1 (3) |
| C4—C5—C6 | 108.2 (2) | O1—C19—C1′ | 125.5 (3) |
| C4—C5—C11 | 107.1 (2) | O2—C19—C1′ | 112.4 (3) |
| C6—C5—C11 | 106.03 (19) | O8—C20—H20A | 109.5 |
| C4—C5—H5 | 111.7 | O8—C20—H20B | 109.5 |
| C6—C5—H5 | 111.7 | H20A—C20—H20B | 109.5 |
| C11—C5—H5 | 111.7 | O8—C20—H20C | 109.5 |
| C5—C6—C7 | 104.6 (2) | H20A—C20—H20C | 109.5 |
| C5—C6—H6A | 110.8 | H20B—C20—H20C | 109.5 |
| C7—C6—H6A | 110.8 | O6—C21—H21A | 109.5 |
| C5—C6—H6B | 110.8 | O6—C21—H21B | 109.5 |
| C7—C6—H6B | 110.8 | H21A—C21—H21B | 109.5 |
| H6A—C6—H6B | 108.9 | O6—C21—H21C | 109.5 |
| C8—C7—C6 | 110.8 (2) | H21A—C21—H21C | 109.5 |
| C8—C7—C17 | 111.3 (2) | H21B—C21—H21C | 109.5 |
| C6—C7—C17 | 103.5 (2) | O7′—C22′—H22D | 109.5 |
| C8—C7—H7 | 110.4 | O7′—C22′—H22E | 109.5 |
| C6—C7—H7 | 110.4 | H22D—C22′—H22E | 109.5 |
| C17—C7—H7 | 110.4 | O7′—C22′—H22F | 109.5 |
| O5—C8—C7 | 106.0 (2) | H22D—C22′—H22F | 109.5 |
| O5—C8—C15 | 107.5 (2) | H22E—C22′—H22F | 109.5 |
| C7—C8—C15 | 111.7 (2) | N2—C23—C24 | 112.3 (3) |
| O5—C8—C9 | 108.5 (2) | N2—C23—H23A | 109.1 |
| C7—C8—C9 | 109.8 (2) | C24—C23—H23A | 109.1 |
| C15—C8—C9 | 113.1 (2) | N2—C23—H23B | 109.1 |
| O4—C9—C14 | 110.7 (2) | C24—C23—H23B | 109.1 |
| O4—C9—C10 | 107.86 (19) | H23A—C23—H23B | 107.9 |
| C14—C9—C10 | 103.6 (2) | C23—C24—H24A | 109.5 |
| O4—C9—C8 | 112.5 (2) | C23—C24—H24B | 109.5 |
| C14—C9—C8 | 109.5 (2) | H24A—C24—H24B | 109.5 |
| C10—C9—C8 | 112.3 (2) | C23—C24—H24C | 109.5 |
| O3—C10—C9 | 106.68 (19) | H24A—C24—H24C | 109.5 |
| O3—C10—C12 | 107.6 (2) | H24B—C24—H24C | 109.5 |
| C9—C10—C12 | 102.42 (19) | C6′—C1′—C2′ | 119.1 (3) |
| O3—C10—C11 | 107.93 (19) | C6′—C1′—C19 | 120.6 (3) |
| C9—C10—C11 | 117.1 (2) | C2′—C1′—C19 | 120.3 (3) |
| C12—C10—C11 | 114.4 (2) | N1—C2′—C3′ | 120.7 (3) |

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|---|---|---|---|
| C17—C11—C1 | 119.2 (2) | N1—C2′—C1′ | 121.8 (3) |
| C17—C11—C5 | 97.83 (19) | C3′—C2′—C1′ | 117.4 (3) |
| C1—C11—C5 | 111.34 (19) | C4′—C3′—C2′ | 122.2 (3) |
| C17—C11—C10 | 107.79 (19) | C4′—C3′—H3′ | 118.9 |
| C1—C11—C10 | 108.1 (2) | C2′—C3′—H3′ | 118.9 |
| C5—C11—C10 | 112.4 (2) | C3′—C4′—C5′ | 120.8 (3) |
| C13—C12—C10 | 107.7 (2) | C3′—C4′—H4′ | 119.6 |
| C13—C12—H12A | 110.2 | C5′—C4′—H4′ | 119.6 |
| C10—C12—H12A | 110.2 | C6′—C5′—C4′ | 118.8 (3) |
| C13—C12—H12B | 110.2 | C6′—C5′—H5′ | 120.6 |
| C10—C12—H12B | 110.2 | C4′—C5′—H5′ | 120.6 |
| H12A—C12—H12B | 108.5 | C5′—C6′—C1′ | 121.7 (3) |
| C16—C13—C14 | 111.9 (3) | C5′—C6′—H6′ | 119.2 |
| C16—C13—C12 | 110.8 (2) | C1′—C6′—H6′ | 119.2 |
| C14—C13—C12 | 101.2 (2) | C2′—N1—H1A | 120.0 |
| C16—C13—H13 | 110.9 | C2′—N1—H1B | 120.0 |
| C14—C13—H13 | 110.9 | H1A—N1—H1B | 120.0 |
| C12—C13—H13 | 110.9 | C18—N2—C23 | 110.7 (2) |
| O6—C14—C13 | 118.6 (2) | C18—N2—C17 | 115.3 (2) |
| O6—C14—C9 | 109.4 (2) | C23—N2—C17 | 113.2 (2) |
| C13—C14—C9 | 101.4 (2) | C19—O2—C4 | 121.5 (2) |
| O6—C14—H14 | 109.0 | C10—O3—H3 | 109.5 |
| C13—C14—H14 | 109.0 | C9—O4—H4 | 109.5 |
| C9—C14—H14 | 109.0 | C8—O5—H5A | 109.5 |
| C16—C15—C8 | 119.7 (2) | C14—O6—C21 | 113.5 (3) |
| C16—C15—H15A | 107.4 | C22—O7—C16 | 110.3 (13) |
| C8—C15—H15A | 107.4 | C22′—O7′—C16 | 142 (2) |
| C16—C15—H15B | 107.4 | C20—O8—C1 | 113.5 (2) |
| | | | |
| O8—C1—C2—C3 | 172.1 (2) | O4—C9—C14—O6 | 70.7 (3) |
| C11—C1—C2—C3 | 46.5 (3) | C10—C9—C14—O6 | −173.9 (2) |
| C1—C2—C3—C4 | −54.4 (3) | C8—C9—C14—O6 | −53.9 (3) |
| C2—C3—C4—O2 | −170.1 (2) | O4—C9—C14—C13 | −163.2 (2) |
| C2—C3—C4—C5 | 66.6 (3) | C10—C9—C14—C13 | −47.9 (2) |
| C2—C3—C4—C18 | −57.7 (3) | C8—C9—C14—C13 | 72.2 (3) |
| O2—C4—C5—C6 | 58.3 (2) | O5—C8—C15—C16 | −136.5 (3) |
| C3—C4—C5—C6 | −178.2 (2) | C7—C8—C15—C16 | 107.7 (3) |
| C18—C4—C5—C6 | −51.7 (3) | C9—C8—C15—C16 | −16.8 (4) |
| O2—C4—C5—C11 | 172.24 (18) | C8—C15—C16—O7′ | 139.0 (12) |
| C3—C4—C5—C11 | −64.2 (3) | C8—C15—C16—O7 | 137.6 (8) |
| C18—C4—C5—C11 | 62.2 (3) | C8—C15—C16—C13 | 17.7 (4) |
| C4—C5—C6—C7 | 100.5 (2) | C14—C13—C16—O7′ | −100.8 (8) |
| C11—C5—C6—C7 | −14.2 (3) | C12—C13—C16—O7′ | 147.2 (8) |
| C5—C6—C7—C8 | 101.8 (2) | C14—C13—C16—O7 | −84.6 (6) |
| C5—C6—C7—C17 | −17.6 (2) | C12—C13—C16—O7 | 163.3 (6) |
| C6—C7—C8—O5 | 64.6 (3) | C14—C13—C16—C15 | 28.1 (3) |
| C17—C7—C8—O5 | 179.14 (19) | C12—C13—C16—C15 | −83.9 (3) |
| C6—C7—C8—C15 | −178.7 (2) | C1—C11—C17—N2 | −48.0 (3) |

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| C17—C7—C8—C15 | −64.1 (3) | C5—C11—C17—N2 | 71.9 (2) |
| C6—C7—C8—C9 | −52.4 (3) | C10—C11—C17—N2 | −171.5 (2) |
| C17—C7—C8—C9 | 62.2 (3) | C1—C11—C17—C7 | −170.4 (2) |
| O5—C8—C9—O4 | −33.9 (3) | C5—C11—C17—C7 | −50.5 (2) |
| C7—C8—C9—O4 | 81.5 (3) | C10—C11—C17—C7 | 66.1 (2) |
| C15—C8—C9—O4 | −153.0 (2) | C8—C7—C17—N2 | 166.20 (19) |
| O5—C8—C9—C14 | 89.7 (2) | C6—C7—C17—N2 | −74.8 (2) |
| C7—C8—C9—C14 | −155.0 (2) | C8—C7—C17—C11 | −75.4 (2) |
| C15—C8—C9—C14 | −29.4 (3) | C6—C7—C17—C11 | 43.5 (2) |
| O5—C8—C9—C10 | −155.8 (2) | O2—C4—C18—N2 | −157.5 (2) |
| C7—C8—C9—C10 | −40.4 (3) | C3—C4—C18—N2 | 84.4 (3) |
| C15—C8—C9—C10 | 85.1 (3) | C5—C4—C18—N2 | −41.5 (3) |
| O4—C9—C10—O3 | 34.5 (3) | O1—C19—C1′—C6′ | −176.2 (3) |
| C14—C9—C10—O3 | −82.8 (2) | O2—C19—C1′—C6′ | 4.5 (4) |
| C8—C9—C10—O3 | 159.04 (19) | O1—C19—C1′—C2′ | 2.0 (5) |
| O4—C9—C10—C12 | 147.5 (2) | O2—C19—C1′—C2′ | −177.3 (3) |
| C14—C9—C10—C12 | 30.1 (2) | C6′—C1′—C2′—N1 | 177.9 (3) |
| C8—C9—C10—C12 | −88.0 (2) | C19—C1′—C2′—N1 | −0.3 (5) |
| O4—C9—C10—C11 | −86.4 (2) | C6′—C1′—C2′—C3′ | −0.7 (4) |
| C14—C9—C10—C11 | 156.2 (2) | C19—C1′—C2′—C3′ | −178.9 (3) |
| C8—C9—C10—C11 | 38.1 (3) | N1—C2′—C3′—C4′ | −177.7 (3) |
| O8—C1—C11—C17 | −55.8 (3) | C1′—C2′—C3′—C4′ | 0.9 (5) |
| C2—C1—C11—C17 | 68.0 (3) | C2′—C3′—C4′—C5′ | −0.2 (5) |
| O8—C1—C11—C5 | −168.5 (2) | C3′—C4′—C5′—C6′ | −0.8 (5) |
| C2—C1—C11—C5 | −44.7 (3) | C4′—C5′—C6′—C1′ | 0.9 (5) |
| O8—C1—C11—C10 | 67.6 (3) | C2′—C1′—C6′—C5′ | −0.2 (5) |
| C2—C1—C11—C10 | −168.6 (2) | C19—C1′—C6′—C5′ | 178.0 (3) |
| C4—C5—C11—C17 | −75.3 (2) | C4—C18—N2—C23 | 169.7 (2) |
| C6—C5—C11—C17 | 40.1 (2) | C4—C18—N2—C17 | 39.6 (3) |
| C4—C5—C11—C1 | 50.3 (3) | C24—C23—N2—C18 | 79.2 (3) |
| C6—C5—C11—C1 | 165.7 (2) | C24—C23—N2—C17 | −149.5 (3) |
| C4—C5—C11—C10 | 171.7 (2) | C11—C17—N2—C18 | −57.3 (3) |
| C6—C5—C11—C10 | −72.9 (2) | C7—C17—N2—C18 | 56.4 (3) |
| O3—C10—C11—C17 | −173.14 (18) | C11—C17—N2—C23 | 173.8 (2) |
| C9—C10—C11—C17 | −52.8 (3) | C7—C17—N2—C23 | −72.5 (3) |
| C12—C10—C11—C17 | 67.1 (3) | O1—C19—O2—C4 | 11.8 (4) |
| O3—C10—C11—C1 | 56.8 (2) | C1′—C19—O2—C4 | −168.9 (2) |
| C9—C10—C11—C1 | 177.13 (19) | C3—C4—O2—C19 | −67.1 (3) |
| C12—C10—C11—C1 | −63.0 (2) | C5—C4—O2—C19 | 57.5 (3) |
| O3—C10—C11—C5 | −66.5 (2) | C18—C4—O2—C19 | 172.8 (2) |
| C9—C10—C11—C5 | 53.9 (3) | C13—C14—O6—C21 | 74.1 (3) |
| C12—C10—C11—C5 | 173.8 (2) | C9—C14—O6—C21 | −170.5 (2) |
| O3—C10—C12—C13 | 110.3 (2) | O7′—C16—O7—C22 | −78 (5) |
| C9—C10—C12—C13 | −1.9 (2) | C15—C16—O7—C22 | 98.3 (9) |
| C11—C10—C12—C13 | −129.7 (2) | C13—C16—O7—C22 | −140.7 (8) |
| C10—C12—C13—C16 | 92.0 (3) | O7—C16—O7′—C22′ | 135 (8) |
| C10—C12—C13—C14 | −26.7 (3) | C15—C16—O7′—C22′ | 130 (3) |
| C16—C13—C14—O6 | 46.8 (3) | C13—C16—O7′—C22′ | −103 (3) |

| | | | |
|---|---|---|---|
| C12—C13—C14—O6 | 164.9 (2) | C2—C1—O8—C20 | 83.5 (3) |
| C16—C13—C14—C9 | −72.9 (3) | C11—C1—O8—C20 | −147.4 (3) |
| C12—C13—C14—C9 | 45.1 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O5—H5*A*···O6 | 0.84 | 2.34 | 2.909 (3) | 126 |
| O4—H4···O5 | 0.84 | 2.27 | 2.684 (3) | 111 |
| O4—H4···O3[i] | 0.84 | 1.94 | 2.713 (3) | 153 |
| O3—H3···O4 | 0.84 | 1.99 | 2.524 (3) | 121 |
| N1—H1*B*···O1 | 0.88 | 2.00 | 2.666 (4) | 131 |
| N1—H1*A*···O8[ii] | 0.88 | 2.19 | 2.999 (3) | 152 |

Symmetry codes: (i) $x+1/2, -y+1/2, -z+2$; (ii) $-x+3/2, -y, z+1/2$.