Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Atalaphylline[1]

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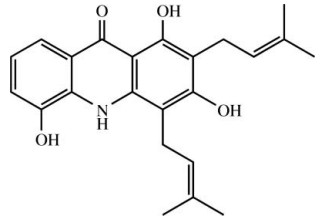
Received 19 November 2009; accepted 2 December 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.118; data-to-parameter ratio = 12.2.

The title acridone alkaloid [systematic name: 1,3,5-trihydroxy-2,4-bis(3-methylbut-2-enyl)acridin-9(10*H*)-one], $C_{23}H_{25}NO_4$, known as atalaphylline, was isolated from *Atalantia monophylla* Corrêa, a mangrove plant. The molecule contains three fused planar rings with an r.m.s. deviation of 0.026 (2) Å. Both 3-methylbut-2-enyl substituents are in a (−)anticlinal conformation. An intramolecular N—H···O hydrogen bond generates an *S*(5) ring motif, while an intramolecular O—H···O hydrogen bond generates an *S*(6) ring motif. In the crystal structure, the molecules are linked into screw chains along [010] by intermolecular O—H···O hydrogen bonds. These chains are stacked along the *a* axis by π–π interactions with centroid–centroid distances of 3.6695 (13) and 3.6696 (13) Å.

Related literature

For hydrogen-bond motifs, see Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987). For details of acridone alkaloids and their biological activity, see: Basu & Basa (1972); Itoigawa *et al.* (2003); Kawaii *et al.* (1999*a*,*b*). For a related structure, see: Chukaew *et al.* (2007). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).



Experimental

Crystal data

$C_{23}H_{25}NO_4$
M_r = 379.44
Orthorhombic, $P2_12_12_1$
a = 5.0650 (1) Å
b = 15.0131 (4) Å
c = 24.5813 (5) Å
V = 1869.20 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.40 × 0.21 × 0.04 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.964, T_{max} = 0.996
17852 measured reflections
3142 independent reflections
2525 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.118
S = 1.03
3142 reflections
257 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O1*···O2	0.82	1.82	2.554 (2)	149
O3—H1*O3*···O2^{i}	0.82	1.93	2.752 (2)	175
N1—H1*N1*···O3	0.86	2.34	2.692 (3)	105

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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* and *PLATON* (Spek, 2009).

The authors thank Prince of Songkla University for financial support through the Crystal Materials Research Unit. NB thanks the Development and Promotion of Science and Technology Talents Project for a fellowship. Mr Arnon Chukaew is acknowledged for supplying the authentic sample of atalaphylline. The authors also thank the Malaysian Government and Universiti Sains Malaysia for the University Golden Goose grant No. 1001/PFIZIK/811012.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2692).

[1] This paper is dedicated to the late His Royal Highness Prince Mahidol of Songkla for his contributions to the development of medical education in Thailand.
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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Basu, D. & Basa, S. C. (1972). *J. Org. Chem*. **37**, 3035–3036.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chukaew, A., Fun, H.-K., Chantrapromma, S. & Ponglimanont, C. (2007). *Acta Cryst.* E**63**, o3723–o3724.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Itoigawa, M., Ito, C., Wu, T. S., Enjo, F., Tokuda, H., Nishino, H. & Furakawa, H. (2003). *Cancer Lett*. **193**, 133–138.

Kawaii, S., Tomono, Y., Katase, E., Ogawa, K., Yano, M., Takemura, Y., Ju-ichi, M. & Furakawa, H. (1999*b*). *Leuk. Res*. **23**, 263–269.

Kawaii, S., Tomono, Y., Katase, E., Ogawa, K., Yano, M., Takemura, Y., Ju-ichi, M., Ito, C. & Furakawa, H. (1999*a*). *J. Nat. Prod*. **62**, 587–589.

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

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2010). E**66**, o81–o82 [doi:10.1107/S1600536809051885]

Atalaphylline

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S1. Comment

Acridone alkaloids display a variety of biological activities such as antiproliferative (Kawii *et al.*, 1999*a*), induction of human promyelocytic leukemia cell (HL-60) differentiation (Kawii *et al.*, 1999*b*) and cancer chemopreventive activities (Itoigawa *et al.*, 2003). The title acridone alkaloid (I) known as atalaphylline (Basu & Basa, 1972) was isolated from *Atalantia monophylla* Corrêa, known locally in Thai as Manao Phi, a mangrove plant which was collected from Trang province in the southern part of Thailand. We previously reported the crystal structure of *N*-methylataphyllinine, an acridone alkaloid which was isolated from the same plant (Chukaew *et al.*, 2007). As part of our research on the crystal structures of natural product compounds from Thai medicinal plants, the molecular and crystal structure of the title acridone alkaloid was investigated and is reported here.

The title molecule (Fig. 1) has a three-fused planar rings with an *r.m.s.* deviation of 0.026 (2) Å. The pyridine ring makes the dihedral angles of 1.82 (11) and 1.14 (11)° with the C1–C3/C11–C13 and C5–C10 benzene rings, respectively. All the three hydroxyl groups are co-planar with the attached benzene rings. All C atoms of each of the 3-methylbut-2-enyl substituents lie on the same plane with *r.m.s.* deviations of 0.018 (2) and 0.004 (3)Å for the C14/C15/C16/C17/C18 and C19/C20/C21/C22/C23 planes, respectively. These two planes make dihedral angles of 88.55 (13) (for the 3-methylbut-2-enyl unit at atom C1) and 69.66 (13)° (for the 3-methylbut-2-enyl unit at atom C12) with the C1–C3/C11–C13 benzene ring. The torsion angles C2–C1–C19–C20 and C13–C12–C14–C15 are -103.0 (3) and -120.8 (2)°, indicating an (-)anti-clinal conformation of both the 3-methylbut-2-enyl units. An intramolecular N—H···O hydrogen bond generates an S(5) ring while an intramolecular O—H···O hydrogen bond generates an S(6) ring motif (Bernstein *et al.*, 1995); these help to maintain the planarity of the acridone skeleton. The bond lengths in (I) are within normal ranges (Allen *et al.*, 1987) and comparable with those found in a related structure (Chukaew *et al.*, 2007).

In the crystal packing (Fig. 2), the molecules are linked into screw chains along the [0 1 0] direction by O3—H1O3···O2 hydrogen bonds (Table 1). These chains are stacked along the *a* axis (Fig. 2) by $\pi\cdots\pi$ interactions with distances $Cg_1\cdots Cg_2$ = 3.6696 (13) Å and $Cg_2\cdots Cg_3$ = 3.6695 (13) Å (symmetry codes for the interactions: 1 + *x*, *y*, *z* and -1 + *x*, *y*, *z* respectively); Cg_1, Cg_2 and Cg_3 are the centroids of C3–C5/C10–C11/N1, C1–C3/C11–C13 and C5–C10 rings, respectively.

S2. Experimental

The air-dried and pulverized root of *A. monophylla* (6.0 kg) was exhaustively extracted with methylene chloride (2 × 20 l for one week) at room temperature. Removal of the solvent from the methylene chloride extract under reduced pressure gave a yellow viscous residue (52.5 g) which was subjected to quick column chromatography over silica gel using solvents of increasing polarity from n-hexane through EtOAc. The eluents were separated into 18 fractions (F1–F18) on the basis of TLC analysis. Fraction F12 (4.3 g) was further separated by quick column chromatography (QCC) with a gradient of acetone-hexane to afford 6 subfractions (12 A-12 F). Subfraction 12 C (385.0 mg) was further purified by

QCC with a gradient of acetone-hexane to give the title compound (22.0 mg). Brown plate-shaped single crystals of the title compound suitable for X-ray structure determination were recrystallized from $CHCl_3/CH_3OH$ (9:1, *v*/*v*) after several days, m.p. 518–520 K.

S3. Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(O—H) = 0.82 Å, d(N—H) = 0.86 Å and d(C—H) = 0.93 Å for aromatic and CH, 0.97 for CH_2 and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for hydroxy and methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.68 Å from C5 and the deepest hole is located at 1.28 Å from C10. A total of 2260 Friedel pairs were merged before final refinement as there is no large anomalous dispersion for the determination of the absolute configuration.

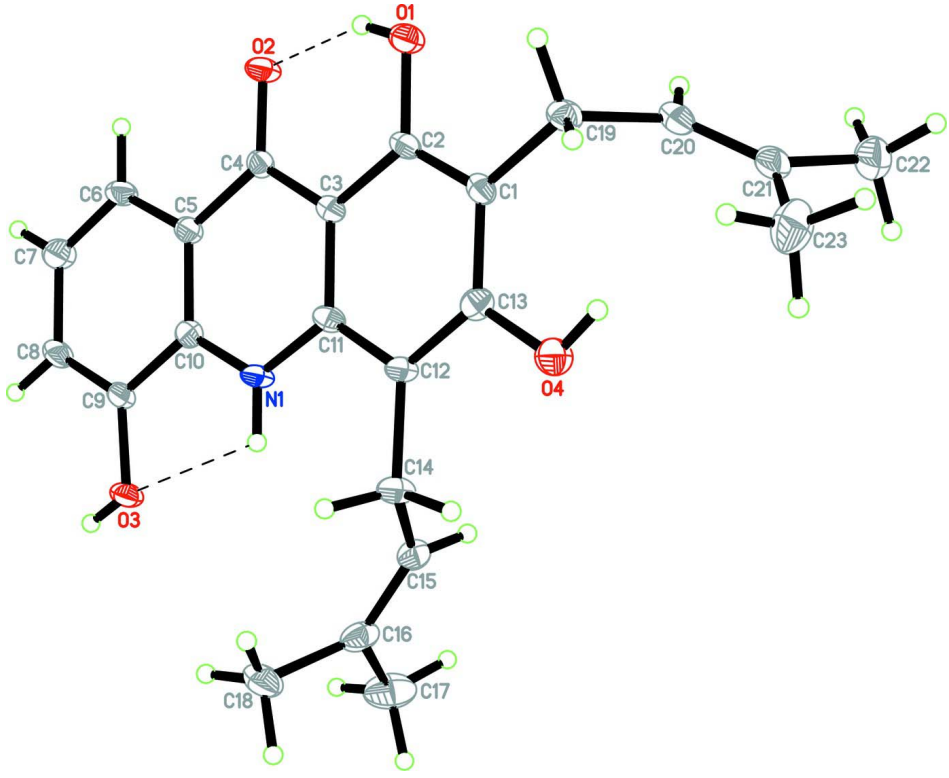

Figure 1

The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atomic numbering scheme. Intramolecular hydrogen bonds are shown as dashed lines.

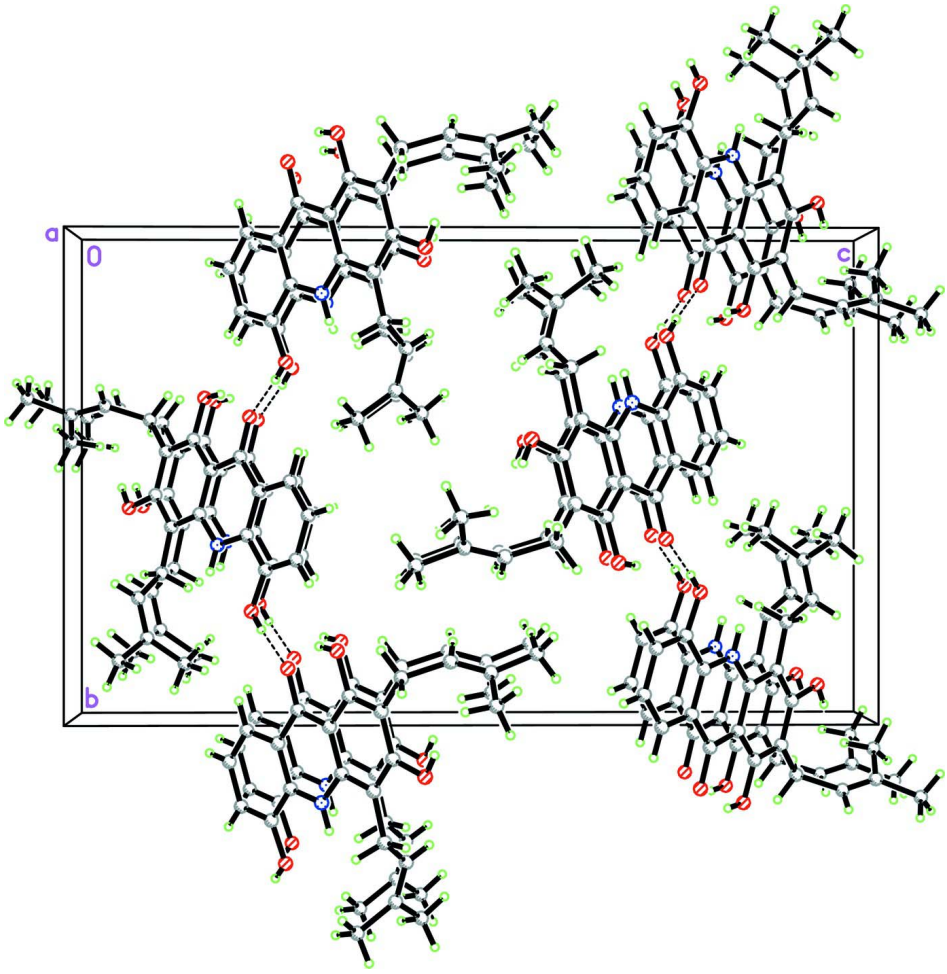

Figure 2

The crystal packing of (I) view along the *a* axis. Hydrogen bonds are drawn as dashed lines.

1,3,5-trihydroxy-2,4-bis(3-methylbut-2-enyl)acridin-9(10*H*)-one

Crystal data

$C_{23}H_{25}NO_4$
$M_r = 379.44$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.0650\ (1)$ Å
$b = 15.0131\ (4)$ Å
$c = 24.5813\ (5)$ Å
$V = 1869.20\ (7)$ Å^3
$Z = 4$
$F(000) = 808$
$D_x = 1.348$ Mg m^{-3}
Melting point = 518–520 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3142 reflections
$\theta = 1.6$–$30.0°$
$\mu = 0.09$ mm^{-1}
$T = 100$ K
Plate, brown
0.40 × 0.21 × 0.04 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.964$, $T_{max} = 0.996$
17852 measured reflections
3142 independent reflections

2525 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 30.0°$, $\theta_{min} = 1.6°$
$h = -6 \rightarrow 7$
$k = -21 \rightarrow 15$
$l = -34 \rightarrow 34$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.118$
$S = 1.03$
3142 reflections
257 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.5624P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.3340 (3)	0.33730 (11)	0.16280 (7)	0.0193 (4)
H1O1	0.4552	0.3307	0.1845	0.029*
O2	0.7218 (3)	0.37946 (11)	0.22404 (6)	0.0183 (4)
O3	0.9035 (4)	0.77231 (10)	0.22808 (6)	0.0187 (4)
H1O3	1.0112	0.8067	0.2415	0.028*
O4	−0.0692 (3)	0.56738 (12)	0.05943 (7)	0.0220 (4)
H1O4	−0.1545	0.5244	0.0487	0.026*
N1	0.6177 (4)	0.64068 (12)	0.18267 (8)	0.0150 (4)
H1N1	0.6002	0.6956	0.1734	0.018*
C1	0.1293 (5)	0.44831 (15)	0.11046 (9)	0.0149 (5)
C2	0.3171 (5)	0.42414 (15)	0.14846 (9)	0.0148 (5)
C3	0.4888 (4)	0.48744 (14)	0.17270 (9)	0.0137 (4)
C4	0.6867 (5)	0.46079 (15)	0.21169 (9)	0.0141 (4)
C5	0.8434 (5)	0.53044 (15)	0.23695 (9)	0.0152 (5)
C6	1.0356 (5)	0.51065 (15)	0.27656 (9)	0.0193 (5)
H6A	1.0648	0.4519	0.2868	0.023*
C7	1.1794 (5)	0.57750 (17)	0.29992 (10)	0.0226 (5)
H7A	1.3061	0.5639	0.3260	0.027*
C8	1.1368 (5)	0.66664 (16)	0.28480 (9)	0.0198 (5)
H8A	1.2342	0.7116	0.3014	0.024*

C9	0.9534 (5)	0.68805 (15)	0.24586 (9)	0.0159 (5)
C10	0.8024 (5)	0.61953 (15)	0.22133 (9)	0.0147 (4)
C11	0.4593 (4)	0.57851 (14)	0.15804 (9)	0.0143 (4)
C12	0.2707 (5)	0.60543 (15)	0.11958 (9)	0.0149 (5)
C13	0.1139 (5)	0.53968 (15)	0.09674 (9)	0.0167 (5)
C14	0.2242 (5)	0.70302 (15)	0.10646 (10)	0.0176 (5)
H14A	0.0747	0.7072	0.0819	0.021*
H14B	0.1765	0.7336	0.1398	0.021*
C15	0.4547 (5)	0.75103 (16)	0.08107 (9)	0.0185 (5)
H15A	0.5447	0.7210	0.0537	0.022*
C16	0.5433 (5)	0.83205 (16)	0.09377 (10)	0.0214 (5)
C17	0.7643 (6)	0.87496 (19)	0.06275 (13)	0.0337 (7)
H17A	0.8238	0.8353	0.0347	0.051*
H17B	0.9077	0.8877	0.0871	0.051*
H17C	0.7028	0.9294	0.0466	0.051*
C18	0.4340 (6)	0.88750 (17)	0.13926 (11)	0.0284 (6)
H18A	0.2903	0.8564	0.1562	0.043*
H18B	0.3717	0.9432	0.1250	0.043*
H18C	0.5700	0.8984	0.1656	0.043*
C19	−0.0597 (5)	0.38065 (15)	0.08666 (9)	0.0179 (5)
H19A	−0.0638	0.3287	0.1101	0.022*
H19B	−0.2358	0.4060	0.0862	0.022*
C20	0.0113 (5)	0.35141 (15)	0.02980 (9)	0.0190 (5)
H20A	0.1620	0.3164	0.0266	0.023*
C21	−0.1154 (5)	0.36960 (16)	−0.01649 (10)	0.0219 (5)
C22	−0.0143 (7)	0.3355 (2)	−0.07035 (10)	0.0323 (7)
H22A	0.1369	0.2982	−0.0643	0.049*
H22B	0.0349	0.3850	−0.0929	0.049*
H22C	−0.1502	0.3017	−0.0880	0.049*
C23	−0.3584 (6)	0.4259 (2)	−0.02118 (12)	0.0333 (6)
H23A	−0.4316	0.4356	0.0143	0.050*
H23B	−0.4859	0.3961	−0.0436	0.050*
H23C	−0.3134	0.4821	−0.0373	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0209 (8)	0.0137 (8)	0.0235 (8)	−0.0020 (7)	−0.0046 (7)	0.0007 (7)
O2	0.0205 (8)	0.0100 (7)	0.0243 (8)	−0.0001 (7)	−0.0030 (7)	0.0012 (7)
O3	0.0236 (9)	0.0098 (7)	0.0227 (8)	−0.0018 (7)	−0.0055 (7)	−0.0001 (6)
O4	0.0206 (8)	0.0205 (8)	0.0251 (9)	−0.0028 (8)	−0.0084 (7)	0.0007 (7)
N1	0.0153 (9)	0.0083 (9)	0.0214 (9)	0.0004 (8)	−0.0029 (8)	0.0009 (7)
C1	0.0155 (10)	0.0137 (10)	0.0155 (10)	−0.0026 (9)	0.0010 (9)	−0.0029 (9)
C2	0.0167 (11)	0.0107 (10)	0.0169 (11)	−0.0015 (9)	0.0031 (9)	−0.0020 (9)
C3	0.0143 (10)	0.0110 (9)	0.0158 (10)	−0.0010 (9)	0.0004 (9)	−0.0014 (8)
C4	0.0147 (10)	0.0133 (10)	0.0141 (10)	0.0009 (9)	−0.0006 (9)	−0.0013 (9)
C5	0.0169 (11)	0.0121 (10)	0.0164 (10)	−0.0003 (9)	0.0003 (9)	−0.0013 (9)
C6	0.0255 (12)	0.0112 (10)	0.0213 (12)	0.0022 (10)	−0.0066 (10)	0.0013 (9)

C7	0.0264 (13)	0.0179 (11)	0.0236 (12)	0.0013 (11)	−0.0108 (11)	0.0011 (10)
C8	0.0246 (12)	0.0135 (11)	0.0211 (12)	−0.0040 (10)	−0.0063 (10)	−0.0031 (9)
C9	0.0194 (11)	0.0113 (10)	0.0170 (11)	−0.0017 (9)	0.0008 (10)	−0.0021 (9)
C10	0.0152 (10)	0.0144 (10)	0.0145 (10)	0.0006 (9)	0.0003 (9)	−0.0003 (9)
C11	0.0125 (10)	0.0115 (9)	0.0190 (11)	−0.0019 (9)	0.0013 (9)	−0.0006 (9)
C12	0.0161 (10)	0.0099 (10)	0.0186 (11)	0.0000 (9)	−0.0008 (9)	0.0011 (9)
C13	0.0159 (11)	0.0176 (11)	0.0167 (10)	0.0012 (10)	0.0006 (9)	0.0014 (9)
C14	0.0154 (11)	0.0121 (10)	0.0252 (12)	−0.0004 (9)	−0.0036 (10)	0.0024 (9)
C15	0.0200 (11)	0.0174 (11)	0.0181 (11)	0.0026 (10)	−0.0021 (9)	0.0023 (9)
C16	0.0192 (11)	0.0191 (11)	0.0258 (12)	−0.0022 (10)	−0.0084 (10)	0.0073 (10)
C17	0.0237 (13)	0.0263 (14)	0.0510 (18)	−0.0056 (13)	−0.0017 (14)	0.0111 (13)
C18	0.0376 (15)	0.0150 (11)	0.0326 (14)	−0.0031 (12)	−0.0083 (13)	−0.0037 (11)
C19	0.0172 (11)	0.0155 (10)	0.0211 (11)	−0.0045 (10)	−0.0004 (9)	0.0005 (9)
C20	0.0202 (11)	0.0148 (10)	0.0220 (12)	−0.0034 (10)	0.0012 (10)	−0.0033 (9)
C21	0.0283 (13)	0.0170 (11)	0.0205 (12)	−0.0085 (11)	0.0017 (11)	−0.0007 (10)
C22	0.0453 (17)	0.0313 (14)	0.0204 (13)	−0.0074 (15)	0.0010 (13)	−0.0022 (11)
C23	0.0268 (14)	0.0446 (17)	0.0287 (14)	−0.0006 (14)	−0.0086 (12)	0.0017 (13)

Geometric parameters (Å, º)

O1—C2	1.353 (3)	C12—C14	1.519 (3)
O1—H1O1	0.8200	C14—C15	1.508 (3)
O2—C4	1.271 (3)	C14—H14A	0.9700
O3—C9	1.362 (3)	C14—H14B	0.9700
O3—H1O3	0.8200	C15—C16	1.334 (3)
O4—C13	1.369 (3)	C15—H15A	0.9300
O4—H1O4	0.8200	C16—C17	1.500 (4)
N1—C10	1.371 (3)	C16—C18	1.500 (4)
N1—C11	1.372 (3)	C17—H17A	0.9600
N1—H1N1	0.8600	C17—H17B	0.9600
C1—C2	1.381 (3)	C17—H17C	0.9600
C1—C13	1.415 (3)	C18—H18A	0.9600
C1—C19	1.514 (3)	C18—H18B	0.9600
C2—C3	1.419 (3)	C18—H18C	0.9600
C3—C11	1.422 (3)	C19—C20	1.508 (3)
C3—C4	1.443 (3)	C19—H19A	0.9700
C4—C5	1.452 (3)	C19—H19B	0.9700
C5—C10	1.407 (3)	C20—C21	1.335 (3)
C5—C6	1.408 (3)	C20—H20A	0.9300
C6—C7	1.366 (3)	C21—C23	1.497 (4)
C6—H6A	0.9300	C21—C22	1.509 (3)
C7—C8	1.406 (3)	C22—H22A	0.9600
C7—H7A	0.9300	C22—H22B	0.9600
C8—C9	1.372 (3)	C22—H22C	0.9600
C8—H8A	0.9300	C23—H23A	0.9600
C9—C10	1.417 (3)	C23—H23B	0.9600
C11—C12	1.404 (3)	C23—H23C	0.9600
C12—C13	1.386 (3)		

C2—O1—H1O1	109.5	C12—C14—H14A	108.4
C9—O3—H1O3	109.5	C15—C14—H14B	108.4
C13—O4—H1O4	109.5	C12—C14—H14B	108.4
C10—N1—C11	123.20 (19)	H14A—C14—H14B	107.5
C10—N1—H1N1	118.4	C16—C15—C14	126.8 (2)
C11—N1—H1N1	118.4	C16—C15—H15A	116.6
C2—C1—C13	117.0 (2)	C14—C15—H15A	116.6
C2—C1—C19	121.4 (2)	C15—C16—C17	121.6 (3)
C13—C1—C19	121.6 (2)	C15—C16—C18	123.8 (2)
O1—C2—C1	118.2 (2)	C17—C16—C18	114.6 (2)
O1—C2—C3	119.8 (2)	C16—C17—H17A	109.5
C1—C2—C3	122.0 (2)	C16—C17—H17B	109.5
C2—C3—C11	118.2 (2)	H17A—C17—H17B	109.5
C2—C3—C4	121.2 (2)	C16—C17—H17C	109.5
C11—C3—C4	120.5 (2)	H17A—C17—H17C	109.5
O2—C4—C3	121.5 (2)	H17B—C17—H17C	109.5
O2—C4—C5	120.9 (2)	C16—C18—H18A	109.5
C3—C4—C5	117.64 (19)	C16—C18—H18B	109.5
C10—C5—C6	119.4 (2)	H18A—C18—H18B	109.5
C10—C5—C4	119.1 (2)	C16—C18—H18C	109.5
C6—C5—C4	121.4 (2)	H18A—C18—H18C	109.5
C7—C6—C5	120.3 (2)	H18B—C18—H18C	109.5
C7—C6—H6A	119.9	C20—C19—C1	113.73 (19)
C5—C6—H6A	119.9	C20—C19—H19A	108.8
C6—C7—C8	120.4 (2)	C1—C19—H19A	108.8
C6—C7—H7A	119.8	C20—C19—H19B	108.8
C8—C7—H7A	119.8	C1—C19—H19B	108.8
C9—C8—C7	120.8 (2)	H19A—C19—H19B	107.7
C9—C8—H8A	119.6	C21—C20—C19	128.0 (2)
C7—C8—H8A	119.6	C21—C20—H20A	116.0
O3—C9—C8	124.5 (2)	C19—C20—H20A	116.0
O3—C9—C10	116.0 (2)	C20—C21—C23	125.2 (2)
C8—C9—C10	119.5 (2)	C20—C21—C22	121.0 (2)
N1—C10—C5	120.7 (2)	C23—C21—C22	113.8 (2)
N1—C10—C9	119.7 (2)	C21—C22—H22A	109.5
C5—C10—C9	119.6 (2)	C21—C22—H22B	109.5
N1—C11—C12	119.97 (19)	H22A—C22—H22B	109.5
N1—C11—C3	118.7 (2)	C21—C22—H22C	109.5
C12—C11—C3	121.3 (2)	H22A—C22—H22C	109.5
C13—C12—C11	117.3 (2)	H22B—C22—H22C	109.5
C13—C12—C14	120.8 (2)	C21—C23—H23A	109.5
C11—C12—C14	121.8 (2)	C21—C23—H23B	109.5
O4—C13—C12	116.3 (2)	H23A—C23—H23B	109.5
O4—C13—C1	119.5 (2)	C21—C23—H23C	109.5
C12—C13—C1	124.2 (2)	H23A—C23—H23C	109.5
C15—C14—C12	115.4 (2)	H23B—C23—H23C	109.5
C15—C14—H14A	108.4		

C13—C1—C2—O1	179.2 (2)	O3—C9—C10—C5	179.3 (2)
C19—C1—C2—O1	2.1 (3)	C8—C9—C10—C5	−0.4 (3)
C13—C1—C2—C3	−0.3 (3)	C10—N1—C11—C12	178.7 (2)
C19—C1—C2—C3	−177.5 (2)	C10—N1—C11—C3	−0.6 (3)
O1—C2—C3—C11	−177.9 (2)	C2—C3—C11—N1	177.7 (2)
C1—C2—C3—C11	1.7 (3)	C4—C3—C11—N1	−1.7 (3)
O1—C2—C3—C4	1.4 (3)	C2—C3—C11—C12	−1.7 (3)
C1—C2—C3—C4	−179.0 (2)	C4—C3—C11—C12	179.0 (2)
C2—C3—C4—O2	3.0 (3)	N1—C11—C12—C13	−179.0 (2)
C11—C3—C4—O2	−177.7 (2)	C3—C11—C12—C13	0.3 (3)
C2—C3—C4—C5	−176.4 (2)	N1—C11—C12—C14	−3.6 (3)
C11—C3—C4—C5	2.9 (3)	C3—C11—C12—C14	175.7 (2)
O2—C4—C5—C10	178.7 (2)	C11—C12—C13—O4	179.85 (19)
C3—C4—C5—C10	−2.0 (3)	C14—C12—C13—O4	4.4 (3)
O2—C4—C5—C6	−1.2 (3)	C11—C12—C13—C1	1.2 (3)
C3—C4—C5—C6	178.2 (2)	C14—C12—C13—C1	−174.3 (2)
C10—C5—C6—C7	0.5 (4)	C2—C1—C13—O4	−179.8 (2)
C4—C5—C6—C7	−179.6 (2)	C19—C1—C13—O4	−2.7 (3)
C5—C6—C7—C8	0.1 (4)	C2—C1—C13—C12	−1.2 (3)
C6—C7—C8—C9	−0.9 (4)	C19—C1—C13—C12	176.0 (2)
C7—C8—C9—O3	−178.7 (2)	C13—C12—C14—C15	−120.8 (2)
C7—C8—C9—C10	1.0 (4)	C11—C12—C14—C15	64.0 (3)
C11—N1—C10—C5	1.6 (3)	C12—C14—C15—C16	−136.1 (2)
C11—N1—C10—C9	−178.3 (2)	C14—C15—C16—C17	−175.8 (2)
C6—C5—C10—N1	179.6 (2)	C14—C15—C16—C18	4.2 (4)
C4—C5—C10—N1	−0.2 (3)	C2—C1—C19—C20	−103.0 (3)
C6—C5—C10—C9	−0.4 (3)	C13—C1—C19—C20	79.9 (3)
C4—C5—C10—C9	179.7 (2)	C1—C19—C20—C21	−110.2 (3)
O3—C9—C10—N1	−0.7 (3)	C19—C20—C21—C23	1.2 (4)
C8—C9—C10—N1	179.6 (2)	C19—C20—C21—C22	179.3 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*1···O2	0.82	1.82	2.554 (2)	149
O3—H1*O*3···O2^{i}	0.82	1.93	2.752 (2)	175
N1—H1*N*1···O3	0.86	2.34	2.692 (3)	105
C14—H14*A*···O4	0.97	2.29	2.773 (3)	110
C19—H19*A*···O1	0.97	2.40	2.811 (3)	105

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