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N-Methylataphyllinine

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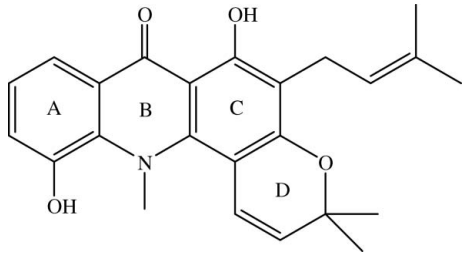
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.129; data-to-parameter ratio = 20.5.

The title acridone alkaloid compound [systematic name: 6,11-dihydroxy-3,3,12-trimethyl-5-(3-methylbut-2-enyl)-3,12-dihydro-7*H*-pyrano[2,3-*c*]acridin-7-one], $C_{24}H_{25}NO_4$, was isolated from *Atalantia monophylla* Corrêa, a mangrove plant. The molecule contains four fused rings. The pyridine ring is in an envelope conformation and the chromene ring adopts a screw-boat conformation. An intramolecular O—H···O hydrogen bond generates an S(6) ring motif. In the crystal structure, the molecules are linked into chains along the b axis by O—H···O hydrogen bonds. These chains are interconnected into molecular sheets parallel to the bc plane by weak C—H···O interactions. The crystal structure is further stabilized by C—H···π interactions.

Related literature

For the biological activities of acridone alkaloids, see: Basa (1975); Basu & Basa (1972); Itoigawa *et al.* (2003); Kawaii *et al.* (1999*a*,*b*). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987). For ring conformations, see: Cremer & Pople (1975). For related structures of non-enzymatic products, see: Chantrapromma, Boonnak & Fun (2005); Chantrapromma, Boonnak, Fun *et al.* (2005); Fun *et al.* (2006); Kosela *et al.* (1999); Pancharoen *et al.* (1984).



Experimental

Crystal data

$C_{24}H_{25}NO_4$
M_r = 391.45
Triclinic, $P\overline{1}$
a = 9.1387 (1) Å
b = 9.7188 (1) Å
c = 11.7006 (2) Å
α = 90.796 (1)°
β = 108.274 (1)°
γ = 100.432 (1)°
V = 967.71 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100.0 (1) K
0.20 × 0.17 × 0.13 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.982, T_{max} = 0.989
27568 measured reflections
5637 independent reflections
4346 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.129
S = 1.06
5637 reflections
275 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

*Cg*1 is the centroid of ring *A* (C5–C10).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*1···O2	0.98 (2)	1.54 (2)	2.4813 (13)	160 (2)
O3—H1*O*3···O1^{i}	0.89 (2)	1.88 (2)	2.7550 (13)	167 (2)
C6—H6*A*···O2^{ii}	0.93	2.55	3.3636 (15)	147
C20—H20*A*···O1	0.97	2.50	2.8976 (15)	105
C18—H18*A*···*Cg*1^{iii}	0.96	2.92	3.3513 (16)	109

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2432).

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supplementary materials

Acta Cryst. (2007). E**63**, o3723-o3724 [**doi:10.1107/S1600536807038238**]

N-Methylataphyllinine

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Comment

Atalantia monophylla Corrêa is a mangrove plant, distributed in South-East Asia and known locally in Thai as Manao Phi. This plant has been used in folk medicine for several purposes such as the treatment of dysentery, chronic rheumatism and paralysis in India (Basa, 1975). Acridone alkaloids had been isolated from the petroleum ether extract of the root bark of this plant (Basu & Basa, 1972). In our continuing interest on the chemical constituents of Thai mangrove plants, we have examined the roots of *A. monophylla* collected from Trang Province in the southern part of Thailand. The title compound was isolated from the methylene chloride extract. It has shown several biological activities such as inhibition of EBV-EA induction (Itoigawa *et al.*, 2003), antiviral (Kawaii *et al.*, 1999*a*) and antiproliferative properties (Kawaii *et al.*, 1999*b*).

The title molecule is chiral, but crystallized in the centrosymmetric space group $P\bar{1}$. This indicates that the crude extract from which the compound was obtained is a racemic mixture and that the compound is a non-enzymatic product (Chantrapromma, Boonnak & Fun, 2005; Chantrapromma, Boonnak, Fun, Anjum *et al.*, 2005; Fun *et al.*, 2006; Kosela *et al.*, 1999; Pancharoen *et al.*, 1984).

The title molecule (Fig. 1) has a four-fused rings (A, B, C and D). The pyridine ring (*B*) is in an envelope conformation, with puckering parameters Q = 0.157 (1) Å, θ = 102.3 (4)° and φ = 170.5 (5)° (Cremer & Pople, 1975), atom N1 having the maximum deviation of 0.102 (1) Å. The chromene ring (D) adopts a screw-boat conformation, with the C14 and O4 atoms deviating from the ring mean plane by 0.276 (11) and −0.238 (1) Å, respectively. The prenyl group (C20–C24) is in a (-)-anticlinal conformation (Fig. 1), as evidenced by the torsion angle C2—C1—C20—C21 of −104.52 (13)°. The dihedral angle between the mean plane of the prenyl unit and the benzene ring *C* is 86.30 (7)°. The two methyl groups are axially and equatorially attached to the ring D at atom C14. The two hydroxyl groups are coplanar with the attached rings.

The bond lengths and angles in the title compound are within normal ranges (Allen *et al.*, 1987).

Intramolecular O1—H1O1···O2 hydrogen bond generates an S(6) ring motif (Bernstein *et al.*, 1995). The molecules are linked into a chain along the *b* axis by O3—H1O3···O1 hydrogen bonds (Fig. 2 and Table 1). These chains are interconnected into molecular sheets by weak C6—H6A···O2 hydrogen bonds (Table 1). In addition, the molecular packing is stabilized by C—H···π interactions involving the ring A (centroid *Cg*1).

Experimental

Air-dried roots of *A. monophylla* (6.0 kg) were ground and extracted with methylene chloride (2 × 20 l) for 7 d at room temperature. The yellow viscous residue (52.5 g) obtained after evaporation of the solvent 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 F6 (1.2 g) was further separated by quick column chromatography (QCC) with a gradient of acetone–hexane to afford eight subfractions (6A-6H). Fraction 6F was further purified by column chromatography (CC) eluting with 20% acetone-hexane to give two subfractions (6FA and 6FB).

Fraction 6FA was recrystallized from acetone to yield brown single crystals of the title compound after several days (m.p. 468–469 K).

Refinement

Hydroxyl H atoms were located in a difference map and isotropically refined. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

Figures

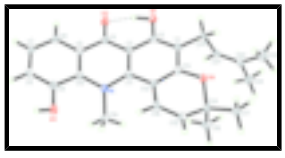

Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering scheme. The hydrogen bond is shown as a dashed line.

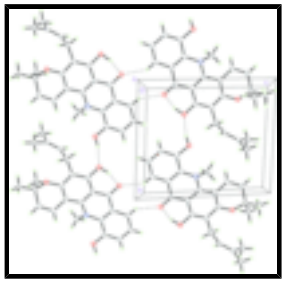

Fig. 2. The crystal packing of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

6,11-dihydroxy-3,3,12-trimethyl-5-(3-methylbut-2-enyl)-3,12-dihydro- 7*H*-pyrano[2,3-*c*]acridin-7-one

Crystal data

$C_{24}H_{25}NO_4$	$Z = 2$
$M_r = 391.45$	$F_{000} = 416$
Triclinic, $P\overline{1}$	$D_x = 1.343$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 468-469 K
$a = 9.1387$ (1) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 9.7188$ (1) Å	Cell parameters from 5637 reflections
$c = 11.7006$ (2) Å	$\theta = 2.1$–30.0º
$\alpha = 90.796$ (1)º	$\mu = 0.09$ mm^{-1}
$\beta = 108.274$ (1)º	$T = 100.0$ (1) K
$\gamma = 100.432$ (1)º	Block, brown
$V = 967.71$ (2) Å^3	0.20 × 0.17 × 0.13 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	5637 independent reflections

Radiation source: fine-focus sealed tube	4346 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.039$
Detector resolution: 8.33 pixels mm^{-1}	$\theta_{max} = 30.0°$
$T = 100.0(1)$ K	$\theta_{min} = 2.1°$
ω scans	$h = -12 \rightarrow 12$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$k = -13 \rightarrow 13$
$T_{min} = 0.982$, $T_{max} = 0.989$	$l = -16 \rightarrow 16$
27568 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.129$	$w = 1/[\sigma^2(F_o^2) + (0.063P)^2 + 0.2437P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.001$
5637 reflections	$\Delta\rho_{max} = 0.47$ e Å^{-3}
275 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The low-temparture data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 $F^2 > 2$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}
O1	0.61622 (11)	0.27676 (9)	0.32200 (8)	0.01883 (19)
H1O1	0.613 (3)	0.222 (2)	0.250 (2)	0.067 (7)*
O2	0.61815 (11)	0.09885 (9)	0.16858 (8)	0.02066 (19)
O3	0.75404 (12)	−0.44307 (10)	0.37072 (8)	0.0228 (2)
H1O3	0.712 (2)	−0.532 (2)	0.3441 (18)	0.049 (6)*
O4	0.70875 (10)	0.15820 (9)	0.72458 (7)	0.01672 (18)
N1	0.76273 (12)	−0.16662 (10)	0.43264 (8)	0.0149 (2)

C1	0.66453 (13)	0.22153 (12)	0.52648 (10)	0.0157 (2)
C2	0.65150 (13)	0.18472 (12)	0.40837 (10)	0.0152 (2)
C3	0.67677 (13)	0.05172 (12)	0.37412 (10)	0.0147 (2)
C4	0.65261 (14)	0.01412 (12)	0.24907 (10)	0.0157 (2)
C5	0.66192 (13)	−0.12935 (12)	0.21856 (10)	0.0160 (2)
C6	0.61480 (14)	−0.17997 (13)	0.09659 (11)	0.0181 (2)
H6A	0.5803	−0.1215	0.0357	0.022*
C7	0.61999 (14)	−0.31638 (13)	0.06789 (11)	0.0198 (2)
H7A	0.5902	−0.3499	−0.0126	0.024*
C8	0.66982 (14)	−0.40457 (13)	0.15928 (11)	0.0190 (2)
H8A	0.6731	−0.4965	0.1389	0.023*
C9	0.71431 (14)	−0.35751 (12)	0.27954 (11)	0.0174 (2)
C10	0.71450 (13)	−0.21600 (12)	0.31109 (10)	0.0151 (2)
C11	0.72382 (13)	−0.04215 (12)	0.46307 (10)	0.0147 (2)
C12	0.73386 (13)	−0.00910 (12)	0.58322 (10)	0.0152 (2)
C13	0.70556 (13)	0.12269 (12)	0.61097 (10)	0.0153 (2)
C14	0.80949 (14)	0.09311 (13)	0.82384 (10)	0.0172 (2)
C15	0.77746 (15)	−0.06229 (13)	0.79078 (11)	0.0190 (2)
H15A	0.7793	−0.1255	0.8499	0.023*
C16	0.74650 (14)	−0.10879 (12)	0.67641 (11)	0.0179 (2)
H16A	0.7330	−0.2041	0.6560	0.021*
C17	0.76250 (15)	0.12599 (14)	0.93288 (11)	0.0210 (3)
H17A	0.6562	0.0791	0.9205	0.032*
H17B	0.7703	0.2254	0.9439	0.032*
H17C	0.8312	0.0943	1.0033	0.032*
C18	0.97943 (15)	0.16100 (15)	0.84190 (12)	0.0242 (3)
H18A	1.0047	0.1423	0.7702	0.036*
H18B	1.0473	0.1230	0.9089	0.036*
H18C	0.9936	0.2605	0.8580	0.036*
C19	0.90568 (14)	−0.20744 (13)	0.51514 (11)	0.0196 (2)
H19A	0.9626	−0.1316	0.5749	0.029*
H19B	0.9711	−0.2282	0.4697	0.029*
H19C	0.8758	−0.2890	0.5542	0.029*
C20	0.64205 (14)	0.36238 (12)	0.56688 (11)	0.0172 (2)
H20A	0.5895	0.4083	0.4970	0.021*
H20B	0.5762	0.3489	0.6182	0.021*
C21	0.79840 (14)	0.45383 (12)	0.63520 (11)	0.0183 (2)
H21A	0.8747	0.4641	0.5972	0.022*
C22	0.83977 (16)	0.52167 (13)	0.74370 (11)	0.0216 (3)
C23	0.73177 (19)	0.51901 (16)	0.81818 (13)	0.0311 (3)
H23A	0.6530	0.4346	0.7960	0.047*
H23B	0.6821	0.5990	0.8039	0.047*
H23C	0.7913	0.5217	0.9022	0.047*
C24	1.00179 (17)	0.60931 (15)	0.79955 (13)	0.0293 (3)
H24A	1.0620	0.6050	0.7460	0.044*
H24B	1.0528	0.5740	0.8749	0.044*
H24C	0.9939	0.7049	0.8134	0.044*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0264 (4)	0.0139 (4)	0.0152 (4)	0.0049 (3)	0.0045 (3)	0.0019 (3)
O2	0.0294 (5)	0.0177 (4)	0.0144 (4)	0.0057 (3)	0.0057 (3)	0.0023 (3)
O3	0.0355 (5)	0.0129 (4)	0.0186 (4)	0.0038 (4)	0.0072 (4)	0.0010 (3)
O4	0.0209 (4)	0.0173 (4)	0.0127 (4)	0.0052 (3)	0.0056 (3)	0.0006 (3)
N1	0.0195 (5)	0.0127 (4)	0.0120 (4)	0.0042 (4)	0.0038 (4)	0.0003 (3)
C1	0.0168 (5)	0.0129 (5)	0.0167 (5)	0.0022 (4)	0.0050 (4)	−0.0002 (4)
C2	0.0164 (5)	0.0127 (5)	0.0153 (5)	0.0018 (4)	0.0040 (4)	0.0023 (4)
C3	0.0167 (5)	0.0133 (5)	0.0136 (5)	0.0017 (4)	0.0048 (4)	0.0001 (4)
C4	0.0169 (5)	0.0152 (5)	0.0142 (5)	0.0024 (4)	0.0041 (4)	0.0011 (4)
C5	0.0173 (5)	0.0157 (5)	0.0151 (5)	0.0020 (4)	0.0059 (4)	−0.0002 (4)
C6	0.0194 (5)	0.0201 (6)	0.0147 (5)	0.0039 (4)	0.0054 (4)	0.0008 (4)
C7	0.0209 (6)	0.0226 (6)	0.0153 (5)	0.0024 (5)	0.0064 (4)	−0.0040 (4)
C8	0.0215 (6)	0.0157 (5)	0.0202 (6)	0.0022 (4)	0.0082 (5)	−0.0032 (4)
C9	0.0197 (5)	0.0152 (5)	0.0180 (6)	0.0030 (4)	0.0071 (4)	0.0010 (4)
C10	0.0160 (5)	0.0154 (5)	0.0139 (5)	0.0013 (4)	0.0059 (4)	−0.0005 (4)
C11	0.0150 (5)	0.0123 (5)	0.0157 (5)	0.0005 (4)	0.0047 (4)	−0.0004 (4)
C12	0.0176 (5)	0.0139 (5)	0.0134 (5)	0.0025 (4)	0.0045 (4)	0.0011 (4)
C13	0.0166 (5)	0.0150 (5)	0.0138 (5)	0.0011 (4)	0.0055 (4)	−0.0004 (4)
C14	0.0191 (5)	0.0186 (6)	0.0133 (5)	0.0038 (4)	0.0044 (4)	0.0015 (4)
C15	0.0245 (6)	0.0176 (6)	0.0165 (5)	0.0059 (5)	0.0076 (5)	0.0040 (4)
C16	0.0233 (6)	0.0135 (5)	0.0175 (6)	0.0040 (4)	0.0074 (5)	0.0020 (4)
C17	0.0254 (6)	0.0224 (6)	0.0155 (5)	0.0045 (5)	0.0070 (5)	−0.0004 (4)
C18	0.0199 (6)	0.0275 (7)	0.0235 (6)	0.0025 (5)	0.0058 (5)	0.0031 (5)
C19	0.0204 (6)	0.0190 (6)	0.0177 (6)	0.0056 (4)	0.0030 (4)	0.0001 (4)
C20	0.0212 (6)	0.0154 (5)	0.0160 (5)	0.0058 (4)	0.0061 (4)	0.0010 (4)
C21	0.0220 (6)	0.0139 (5)	0.0202 (6)	0.0047 (4)	0.0078 (5)	0.0012 (4)
C22	0.0278 (6)	0.0165 (6)	0.0198 (6)	0.0071 (5)	0.0050 (5)	0.0009 (4)
C23	0.0442 (8)	0.0300 (7)	0.0224 (7)	0.0092 (6)	0.0145 (6)	−0.0027 (5)
C24	0.0314 (7)	0.0223 (7)	0.0268 (7)	0.0057 (5)	−0.0011 (6)	−0.0044 (5)

Geometric parameters (Å, °)

O1—C2	1.3604 (14)	C14—C15	1.5064 (17)
O1—H1O1	0.98 (2)	C14—C17	1.5153 (16)
O2—C4	1.2624 (14)	C14—C18	1.5203 (17)
O3—C9	1.3610 (15)	C15—C16	1.3321 (16)
O3—H1O3	0.88 (2)	C15—H15A	0.93
O4—C13	1.3588 (13)	C16—H16A	0.93
O4—C14	1.4688 (14)	C17—H17A	0.96
N1—C11	1.3927 (14)	C17—H17B	0.96
N1—C10	1.3995 (14)	C17—H17C	0.96
N1—C19	1.4844 (15)	C18—H18A	0.96
C1—C2	1.3855 (16)	C18—H18B	0.96
C1—C13	1.4019 (16)	C18—H18C	0.96
C1—C20	1.5113 (16)	C19—H19A	0.96

C2—C3	1.4271 (16)	C19—H19B	0.96
C3—C11	1.4099 (16)	C19—H19C	0.96
C3—C4	1.4410 (15)	C20—C21	1.5082 (17)
C4—C5	1.4585 (16)	C20—H20A	0.97
C5—C10	1.3993 (17)	C20—H20B	0.97
C5—C6	1.4079 (16)	C21—C22	1.3323 (17)
C6—C7	1.3758 (17)	C21—H21A	0.93
C6—H6A	0.93	C22—C23	1.5050 (19)
C7—C8	1.3953 (18)	C22—C24	1.5053 (19)
C7—H7A	0.93	C23—H23A	0.96
C8—C9	1.3823 (16)	C23—H23B	0.96
C8—H8A	0.93	C23—H23C	0.96
C9—C10	1.4186 (16)	C24—H24A	0.96
C11—C12	1.4085 (15)	C24—H24B	0.96
C12—C13	1.4049 (16)	C24—H24C	0.96
C12—C16	1.4601 (16)		
C2—O1—H1O1	101.5 (13)	C17—C14—C18	111.13 (10)
C9—O3—H1O3	110.2 (13)	C16—C15—C14	119.69 (11)
C13—O4—C14	117.12 (9)	C16—C15—H15A	120.2
C11—N1—C10	119.09 (10)	C14—C15—H15A	120.2
C11—N1—C19	117.83 (9)	C15—C16—C12	119.58 (11)
C10—N1—C19	117.58 (9)	C15—C16—H16A	120.2
C2—C1—C13	116.87 (10)	C12—C16—H16A	120.2
C2—C1—C20	123.15 (11)	C14—C17—H17A	109.5
C13—C1—C20	119.95 (10)	C14—C17—H17B	109.5
O1—C2—C1	120.20 (10)	H17A—C17—H17B	109.5
O1—C2—C3	118.25 (10)	C14—C17—H17C	109.5
C1—C2—C3	121.54 (11)	H17A—C17—H17C	109.5
C11—C3—C2	119.55 (10)	H17B—C17—H17C	109.5
C11—C3—C4	120.67 (10)	C14—C18—H18A	109.5
C2—C3—C4	119.77 (11)	C14—C18—H18B	109.5
O2—C4—C3	121.89 (10)	H18A—C18—H18B	109.5
O2—C4—C5	121.25 (10)	C14—C18—H18C	109.5
C3—C4—C5	116.79 (10)	H18A—C18—H18C	109.5
C10—C5—C6	120.80 (11)	H18B—C18—H18C	109.5
C10—C5—C4	119.49 (10)	N1—C19—H19A	109.5
C6—C5—C4	119.70 (11)	N1—C19—H19B	109.5
C7—C6—C5	119.68 (12)	H19A—C19—H19B	109.5
C7—C6—H6A	120.2	N1—C19—H19C	109.5
C5—C6—H6A	120.2	H19A—C19—H19C	109.5
C6—C7—C8	120.11 (11)	H19B—C19—H19C	109.5
C6—C7—H7A	119.9	C21—C20—C1	110.51 (10)
C8—C7—H7A	119.9	C21—C20—H20A	109.5
C9—C8—C7	121.10 (11)	C1—C20—H20A	109.5
C9—C8—H8A	119.5	C21—C20—H20B	109.5
C7—C8—H8A	119.5	C1—C20—H20B	109.5
O3—C9—C8	122.58 (11)	H20A—C20—H20B	108.1
O3—C9—C10	117.72 (10)	C22—C21—C20	127.50 (12)
C8—C9—C10	119.67 (11)	C22—C21—H21A	116.3

C5—C10—N1	121.34 (10)	C20—C21—H21A	116.3
C5—C10—C9	118.58 (10)	C21—C22—C23	123.88 (12)
N1—C10—C9	120.06 (11)	C21—C22—C24	120.84 (12)
N1—C11—C12	119.91 (10)	C23—C22—C24	115.27 (11)
N1—C11—C3	120.07 (10)	C22—C23—H23A	109.5
C12—C11—C3	120.02 (10)	C22—C23—H23B	109.5
C13—C12—C11	117.58 (11)	H23A—C23—H23B	109.5
C13—C12—C16	116.87 (10)	C22—C23—H23C	109.5
C11—C12—C16	124.79 (10)	H23A—C23—H23C	109.5
O4—C13—C1	115.59 (10)	H23B—C23—H23C	109.5
O4—C13—C12	120.03 (10)	C22—C24—H24A	109.5
C1—C13—C12	124.29 (10)	C22—C24—H24B	109.5
O4—C14—C15	108.29 (9)	H24A—C24—H24B	109.5
O4—C14—C17	105.00 (9)	C22—C24—H24C	109.5
C15—C14—C17	112.06 (10)	H24A—C24—H24C	109.5
O4—C14—C18	108.01 (10)	H24B—C24—H24C	109.5
C15—C14—C18	111.99 (10)		
C13—C1—C2—O1	−178.24 (10)	C19—N1—C11—C12	−43.29 (15)
C20—C1—C2—O1	−0.31 (17)	C10—N1—C11—C3	−17.49 (16)
C13—C1—C2—C3	0.75 (17)	C19—N1—C11—C3	135.73 (11)
C20—C1—C2—C3	178.68 (10)	C2—C3—C11—N1	−174.13 (10)
O1—C2—C3—C11	175.77 (10)	C4—C3—C11—N1	6.46 (16)
C1—C2—C3—C11	−3.23 (17)	C2—C3—C11—C12	4.89 (16)
O1—C2—C3—C4	−4.81 (16)	C4—C3—C11—C12	−174.52 (10)
C1—C2—C3—C4	176.18 (10)	N1—C11—C12—C13	174.99 (10)
C11—C3—C4—O2	−176.40 (11)	C3—C11—C12—C13	−4.03 (16)
C2—C3—C4—O2	4.19 (17)	N1—C11—C12—C16	−15.31 (17)
C11—C3—C4—C5	6.65 (16)	C3—C11—C12—C16	165.67 (11)
C2—C3—C4—C5	−172.77 (10)	C14—O4—C13—C1	−155.25 (10)
O2—C4—C5—C10	174.13 (11)	C14—O4—C13—C12	28.13 (15)
C3—C4—C5—C10	−8.89 (16)	C2—C1—C13—O4	−176.40 (10)
O2—C4—C5—C6	−7.23 (17)	C20—C1—C13—O4	5.61 (15)
C3—C4—C5—C6	169.75 (10)	C2—C1—C13—C12	0.07 (17)
C10—C5—C6—C7	0.20 (18)	C20—C1—C13—C12	−177.93 (11)
C4—C5—C6—C7	−178.42 (11)	C11—C12—C13—O4	177.89 (10)
C5—C6—C7—C8	0.82 (18)	C16—C12—C13—O4	7.37 (16)
C6—C7—C8—C9	0.16 (18)	C11—C12—C13—C1	1.57 (17)
C7—C8—C9—O3	175.78 (11)	C16—C12—C13—C1	−168.95 (11)
C7—C8—C9—C10	−2.12 (18)	C13—O4—C14—C15	−48.10 (13)
C6—C5—C10—N1	179.53 (10)	C13—O4—C14—C17	−167.97 (9)
C4—C5—C10—N1	−1.85 (17)	C13—O4—C14—C18	73.37 (12)
C6—C5—C10—C9	−2.12 (17)	O4—C14—C15—C16	36.03 (15)
C4—C5—C10—C9	176.51 (10)	C17—C14—C15—C16	151.37 (12)
C11—N1—C10—C5	15.26 (16)	C18—C14—C15—C16	−82.95 (14)
C19—N1—C10—C5	−138.02 (11)	C14—C15—C16—C12	−3.98 (18)
C11—N1—C10—C9	−163.07 (10)	C13—C12—C16—C15	−19.66 (17)
C19—N1—C10—C9	43.65 (15)	C11—C12—C16—C15	170.58 (12)
O3—C9—C10—C5	−174.94 (10)	C2—C1—C20—C21	−104.52 (13)
C8—C9—C10—C5	3.06 (17)	C13—C1—C20—C21	73.35 (14)

O3—C9—C10—N1	3.43 (16)	C1—C20—C21—C22	−128.12 (13)
C8—C9—C10—N1	−178.57 (11)	C20—C21—C22—C23	−0.7 (2)
C10—N1—C11—C12	163.49 (10)	C20—C21—C22—C24	−179.97 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O1···O2	0.98 (2)	1.54 (2)	2.4813 (13)	160 (2)
O3—H1O3···O1^{i}	0.89 (2)	1.88 (2)	2.7550 (13)	167 (2)
C6—H6A···O2^{ii}	0.93	2.55	3.3636 (15)	147
C20—H20A···O1	0.97	2.50	2.8976 (15)	105
C18—H18A···Cg1^{iii}	0.96	2.92	3.3513 (16)	109

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

Fig. 1

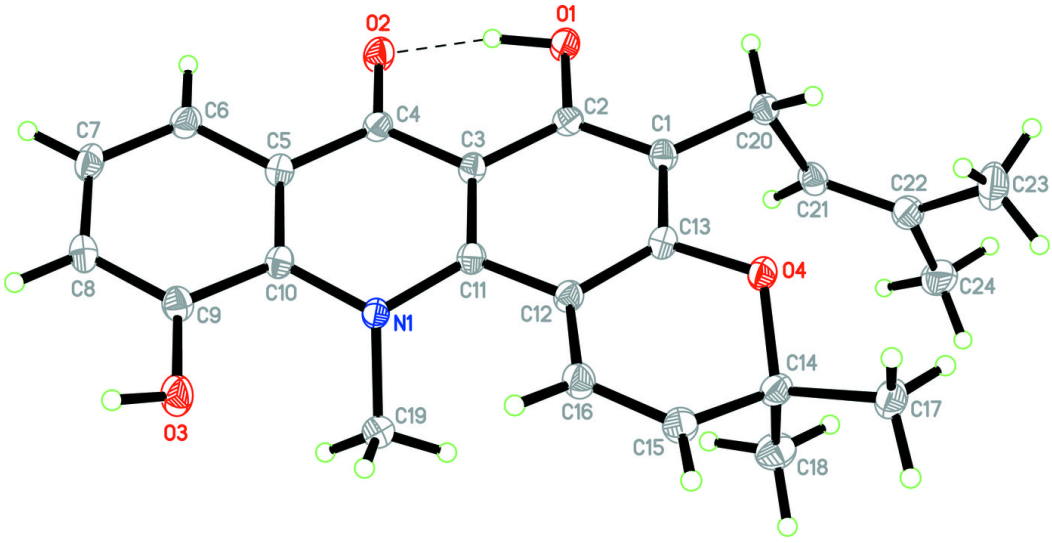
O2
O1
C6
C7
C4
C5
C3
C2
C1
C20
C21
C22
C23
C13
O4
C8
C10
C11
C12
C24
C9
N1
C14
C19
C16
C17
O3
C15
C18

Fig. 2