Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 1′-(2-hydroxy-1,3-dioxoindan-2-yl)-1,3-dioxoindane-2-spiro-3′-pyrrolizidine-1′-carboxylate

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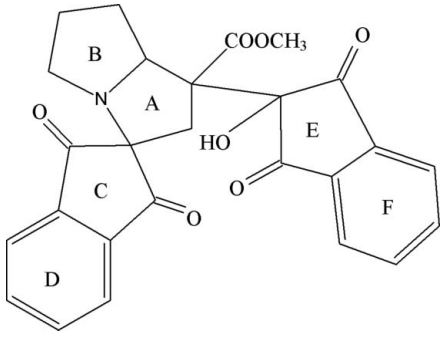
Received 6 July 2007; accepted 20 July 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.049; wR factor = 0.169; data-to-parameter ratio = 12.9.

In the title compound, $C_{26}H_{21}NO_7$, all the five-membered rings adopt envelope conformations. The pyrrolizine nucleus is folded about the shared N—C bond. The molecule is stabilized by an intramolecular O—H···O hydrogen bond which generates an S(8) motif. The crystal packing reveals that symmetry-related molecules are linked into a three-dimensional network by C—H···O hydrogen bonds.

Related literature

For general background, see: Amalraj *et al.* (2003); Cordell (1981); Suzuki *et al.* (1994). For synthesis, see: Ramesh *et al.* (2007). For ring conformations, see: Cremer & Pople (1975); Nardelli (1983). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{26}H_{21}NO_7$
M_r = 459.44
Monoclinic, $C2/c$
a = 39.580 (5) Å
b = 7.6690 (15) Å
c = 14.868 (3) Å
β = 103.291 (16)°
V = 4392.0 (14) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 0.85 mm^{-1}
T = 293 (2) K
0.37 × 0.24 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.784, T_{max} = 0.881
4055 measured reflections
3994 independent reflections
3118 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
2 standard reflections
frequency: 60 min
intensity decay: 4%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.169
S = 0.99
3994 reflections
309 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O4	0.82	1.89	2.691 (2)	165
C8—H8A···O4^{i}	0.97	2.56	3.259 (3)	130
C16—H16···O3^{ii}	0.93	2.57	3.481 (3)	168
C22—H22···O7^{iii}	0.93	2.38	3.280 (4)	163
C23—H23···O1^{iv}	0.93	2.47	3.238 (3)	140

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1988); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

SM thanks Dr Babu Varghese, SAIF, IIT, Chennai, India, for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2413).

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  doi:10.1107/S160053680703560X

supplementary materials

Acta Cryst. (2007). E**63**, o3610 [**doi:10.1107/S160053680703560X**]

Methyl 1'-(2-hydroxy-1,3-dioxoindan-2-yl)-1,3-dioxoindane-2-spiro-3'-pyrrolizidine-1'-carboxylate

S. Murugavel, P. Ramesh, A. SubbiahPandi, E. Ramesh and R. Raghunathan

Comment

Pyrrolizidine alkaloids represent a group of compounds present in a variety of plants throughout the world. The pyrrolizidine alkaloids are well documented for their mutagenic, antineoplastic, carcinogenic, hepatoxic and many pharmacological activities. Substituted pyrrolidine compounds have been found to have antimicrobial and antifungal activity against various pathogens (Amalraj *et al.*, 2003). Several optically active pyrrolidine compounds have been used as intermediates in controlled asymmetric synthesis (Suzuki *et al.*, 1994). The spiro-indole-pyrrolidine ring system is a frequently encountered structural motif in many biologically important and pharmacologically relevant alkaloids, *e.g.* vincrinstine, vinblastine and Spirotypostatins (Cordell, 1981). Against this background and in order to obtain detailed information on its molecular conformation, the structure determination of the title compound has been carried out and the results are presented here.

The title compound (Fig.1) consists of a pyrrolizine ring system (rings A and B) connected to a indane dione group (rings C and D) at C2, hydroxyl indane dione group (rings E and F) and methoxy carbonyl group at C4. All the five-membered rings A, B, C and E adopt envelope conformations. The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) for the pyrrolidine ring A are q_2 = 0.245 (2) Å, φ = 187.8 (5)° and Δ_s(N1) = 4.7 (2)°, for the pyrrolidine ring B are q_2 = 0.399 (2) Å, φ = 318.6 (4)° and Δ_s(C8) = 3.2 (2)°, for the ring C are q_2 = 0.141 (2) Å, φ = 5.0 (10)° and Δ_s(C2) = 1.7 (3)°, and for the ring E are q_2 = 0.090 (3) Å, φ = 356.8 (17)° and Δ_s(C19) = 0.7 (3)°.

The molecule is stabilized by the intramolecular O5—H5···O4 hydrogen bond which generates an S(8) motif (Bernstein *et al.*, 1995). The crystal packing is stabilized by intermolecular C—H···O hydrogen bonds. Atom C22 in the molecule at (*x*, *y*, *z*) donates one proton to atom O7 in the molecule at (*x*, 1 + *y*, *z*), forming a C(6) chain along the *b* axis. Also, atoms C8 and C16 in the molecule at (*x*, *y*, *z*) donate one proton each to atom O4 and O3 in the molecule at (*x*, 1 − *y*, 1/2 + *z*) and (*x*, 1 − *y*, −1/2 + *z*), respectively, forming a chain along the *c* axis. These hydrogen bonds generate an $R^2_2(11)$ ring motif. The molecules at (*x*, *y*, *z*) and (−*x*, 2 − *y*, −*z*) are linked by C23—H23···O1 hydrogen bonds into cyclic centrosymmetric $R^2_2(18)$ dimers. Thus, the symmetry-related molecules are cross-linked by these hydrogen bonds to generate a three-dimensional network.

Experimental

The title compound was synthesized according to the method reported in the literature (Ramesh *et al.*, 2007). A solution of the Baylis–Hilman adduct of ninhydrin (1 mmol), proline (1 mmol) and ninhydrin (1 mmol) in methonol was refluxed. After completion of the reaction, as monitored by thin-layer chromatographic analysis, the solvent was removed under vacuum, and the crude product was subjected to column chromatography on silica gel (100–200 mesh) using petroleum ether/ethyl acetate (7:3) as eluent. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a ethanol solution.

Refinement

H atoms were positioned geometrically (O—H = 0.82 Å and C—H = 0.93–0.98 Å) and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C,*O*) for the methyl and OH groups.

Figures

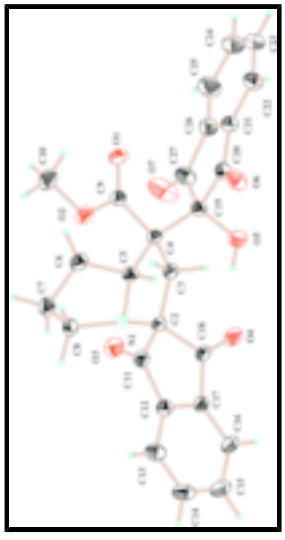


Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Methyl 1'-(2-hydroxy-1,3-dioxoindan-2-yl)-1,3-dioxoindane-2- spiro-3'-pyrrolizidine-1-carboxylate

Crystal data

$C_{26}H_{21}NO_7$	F_{000} = 1920
M_r = 459.44	D_x = 1.390 Mg m^{-3}
Monoclinic, *C*2/*c*	Cu *K*α radiation λ = 1.54180 Å
Hall symbol: -C 2yc	Cell parameters from 25 reflections
a = 39.580 (5) Å	θ = 2.3–68.0º
b = 7.6690 (15) Å	μ = 0.85 mm^{-1}
c = 14.868 (3) Å	*T* = 293 (2) K
β = 103.291 (16)º	Block, colourless
V = 4392.0 (14) Å^3	0.37 × 0.24 × 0.15 mm
Z = 8	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.052
Radiation source: fine-focus sealed tube	θ_{max} = 68.0º
Monochromator: graphite	θ_{min} = 2.3º
T = 293(2) K	*h* = 0→47

ω/2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.784, T_{max} = 0.881
4055 measured reflections
3994 independent reflections
3118 reflections with $I > 2\sigma(I)$

$k = 0 \rightarrow 9$
$l = -17 \rightarrow 17$
2 standard reflections
every 60 min
intensity decay: 4%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.169$
$S = 0.99$
3994 reflections
309 parameters
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
$w = 1/[\sigma^2(F_o^2) + (0.1055P)^2 + 3.9632P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00074 (11)

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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.07308 (5)	0.7325 (3)	0.11713 (13)	0.0797 (7)
O2	0.12466 (4)	0.7130 (2)	0.21385 (11)	0.0554 (4)
O3	0.22421 (4)	0.6868 (3)	0.21202 (11)	0.0619 (5)
O4	0.17375 (4)	0.5356 (3)	−0.09529 (11)	0.0693 (6)
O5	0.10878 (5)	0.6593 (3)	−0.11651 (11)	0.0674 (6)
H5	0.1292	0.6292	−0.1003	0.101*
O6	0.10669 (5)	1.0111 (2)	−0.03087 (14)	0.0685 (5)
O7	0.05550 (6)	0.4526 (3)	−0.0507 (2)	0.1019 (9)
N1	0.16725 (4)	0.4251 (2)	0.09413 (11)	0.0388 (4)

C2	0.18367 (5)	0.5860 (3)	0.07124 (13)	0.0387 (5)
C3	0.15739 (5)	0.7337 (3)	0.06648 (15)	0.0404 (5)
H3A	0.1577	0.8093	0.0144	0.048*
H3B	0.1629	0.8027	0.1226	0.048*
C4	0.12111 (5)	0.6473 (3)	0.05505 (13)	0.0379 (5)
C5	0.12937 (5)	0.4442 (3)	0.06061 (15)	0.0401 (5)
H5A	0.1227	0.3958	−0.0020	0.048*
C6	0.11382 (6)	0.3280 (3)	0.12542 (18)	0.0537 (6)
H6A	0.1010	0.3976	0.1608	0.064*
H6B	0.0983	0.2414	0.0904	0.064*
C7	0.14515 (7)	0.2410 (3)	0.18904 (18)	0.0582 (6)
H7A	0.1412	0.2232	0.2504	0.070*
H7B	0.1502	0.1295	0.1642	0.070*
C8	0.17429 (6)	0.3687 (3)	0.19145 (15)	0.0506 (6)
H8A	0.1731	0.4658	0.2325	0.061*
H8B	0.1968	0.3129	0.2103	0.061*
C9	0.10343 (5)	0.7037 (3)	0.13072 (15)	0.0438 (5)
C10	0.10900 (8)	0.7620 (4)	0.28863 (19)	0.0719 (8)
H10A	0.0999	0.8781	0.2785	0.108*
H10B	0.1262	0.7580	0.3459	0.108*
H10C	0.0905	0.6824	0.2913	0.108*
C11	0.21928 (6)	0.6158 (3)	0.13721 (15)	0.0444 (5)
C12	0.24625 (5)	0.5492 (3)	0.09159 (15)	0.0433 (5)
C13	0.28137 (6)	0.5255 (3)	0.1270 (2)	0.0581 (6)
H13	0.2915	0.5499	0.1885	0.070*
C14	0.30097 (6)	0.4644 (4)	0.0679 (2)	0.0663 (7)
H14	0.3247	0.4472	0.0903	0.080*
C15	0.28613 (7)	0.4280 (3)	−0.0240 (2)	0.0603 (7)
H15	0.3000	0.3872	−0.0621	0.072*
C16	0.25130 (6)	0.4510 (3)	−0.05965 (18)	0.0519 (6)
H16	0.2414	0.4279	−0.1215	0.062*
C17	0.23135 (5)	0.5101 (3)	−0.00032 (15)	0.0417 (5)
C18	0.19387 (5)	0.5440 (3)	−0.01998 (14)	0.0429 (5)
C19	0.09585 (5)	0.6972 (3)	−0.03765 (15)	0.0432 (5)
C20	0.08603 (6)	0.8913 (3)	−0.04602 (15)	0.0461 (5)
C21	0.04821 (6)	0.9050 (3)	−0.08045 (16)	0.0509 (6)
C22	0.02816 (8)	1.0539 (4)	−0.1044 (2)	0.0666 (7)
H22	0.0382	1.1641	−0.0999	0.080*
C23	−0.00700 (8)	1.0321 (5)	−0.1347 (2)	0.0837 (10)
H23	−0.0210	1.1295	−0.1517	0.100*
C24	−0.02206 (8)	0.8695 (6)	−0.1408 (3)	0.0916 (11)
H24	−0.0460	0.8592	−0.1618	0.110*
C25	−0.00228 (7)	0.7217 (5)	−0.1162 (2)	0.0813 (9)
H25	−0.0125	0.6120	−0.1196	0.098*
C26	0.03318 (6)	0.7415 (4)	−0.08649 (18)	0.0572 (6)
C27	0.06007 (6)	0.6068 (3)	−0.05542 (19)	0.0596 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0564 (11)	0.1316 (19)	0.0562 (11)	0.0390 (11)	0.0233 (8)	0.0113 (11)
O2	0.0560 (9)	0.0683 (11)	0.0452 (9)	0.0027 (8)	0.0184 (7)	−0.0102 (8)
O3	0.0600 (10)	0.0793 (12)	0.0442 (9)	−0.0009 (9)	0.0073 (7)	−0.0162 (8)
O4	0.0507 (9)	0.1217 (17)	0.0359 (9)	0.0137 (10)	0.0108 (7)	−0.0045 (9)
O5	0.0641 (11)	0.0972 (15)	0.0397 (9)	0.0275 (10)	0.0093 (7)	−0.0066 (9)
O6	0.0660 (11)	0.0474 (10)	0.0853 (14)	−0.0032 (8)	0.0033 (10)	0.0134 (9)
O7	0.0690 (13)	0.0490 (12)	0.166 (3)	−0.0109 (10)	−0.0172 (14)	0.0075 (13)
N1	0.0420 (9)	0.0408 (9)	0.0358 (9)	0.0042 (7)	0.0136 (7)	0.0006 (7)
C2	0.0387 (10)	0.0439 (11)	0.0362 (10)	0.0013 (8)	0.0139 (8)	0.0003 (8)
C3	0.0422 (11)	0.0379 (11)	0.0436 (11)	0.0000 (8)	0.0151 (8)	−0.0009 (9)
C4	0.0384 (10)	0.0381 (11)	0.0390 (11)	0.0030 (8)	0.0128 (8)	0.0003 (8)
C5	0.0421 (11)	0.0353 (10)	0.0451 (11)	0.0015 (8)	0.0146 (8)	−0.0030 (8)
C6	0.0578 (14)	0.0404 (12)	0.0708 (16)	−0.0016 (10)	0.0307 (12)	0.0032 (11)
C7	0.0731 (16)	0.0492 (13)	0.0587 (14)	0.0032 (12)	0.0281 (12)	0.0118 (11)
C8	0.0598 (13)	0.0521 (13)	0.0414 (11)	0.0080 (11)	0.0151 (10)	0.0081 (10)
C9	0.0462 (12)	0.0416 (12)	0.0472 (12)	0.0082 (9)	0.0179 (9)	0.0033 (9)
C10	0.090 (2)	0.0815 (19)	0.0528 (15)	0.0068 (16)	0.0352 (14)	−0.0064 (14)
C11	0.0455 (11)	0.0468 (12)	0.0411 (11)	0.0001 (9)	0.0106 (9)	−0.0008 (9)
C12	0.0415 (11)	0.0397 (11)	0.0497 (12)	−0.0023 (9)	0.0128 (9)	0.0004 (9)
C13	0.0409 (12)	0.0566 (15)	0.0734 (17)	−0.0021 (10)	0.0061 (11)	−0.0038 (12)
C14	0.0386 (12)	0.0605 (16)	0.102 (2)	0.0028 (11)	0.0202 (13)	0.0014 (15)
C15	0.0531 (14)	0.0488 (13)	0.090 (2)	0.0010 (11)	0.0390 (13)	−0.0045 (13)
C16	0.0556 (13)	0.0488 (13)	0.0601 (14)	−0.0022 (10)	0.0317 (11)	−0.0023 (10)
C17	0.0437 (11)	0.0392 (11)	0.0462 (11)	−0.0008 (8)	0.0184 (9)	0.0019 (9)
C18	0.0427 (11)	0.0526 (12)	0.0358 (11)	0.0009 (9)	0.0139 (9)	0.0011 (9)
C19	0.0431 (11)	0.0439 (12)	0.0431 (11)	0.0060 (9)	0.0113 (9)	−0.0026 (9)
C20	0.0502 (12)	0.0463 (12)	0.0418 (11)	0.0024 (10)	0.0103 (9)	0.0040 (9)
C21	0.0505 (13)	0.0565 (14)	0.0469 (12)	0.0122 (10)	0.0136 (10)	0.0036 (10)
C22	0.0699 (17)	0.0660 (17)	0.0646 (16)	0.0251 (14)	0.0170 (13)	0.0096 (13)
C23	0.0645 (18)	0.104 (3)	0.088 (2)	0.0416 (18)	0.0273 (16)	0.0277 (19)
C24	0.0455 (15)	0.129 (3)	0.101 (2)	0.0209 (18)	0.0179 (15)	0.034 (2)
C25	0.0469 (14)	0.094 (2)	0.098 (2)	−0.0011 (15)	0.0061 (14)	0.0195 (19)
C26	0.0462 (12)	0.0637 (16)	0.0612 (15)	0.0041 (11)	0.0112 (11)	0.0069 (12)
C27	0.0494 (13)	0.0533 (15)	0.0696 (16)	−0.0003 (11)	0.0007 (11)	0.0003 (12)

Geometric parameters (Å, °)

O1—C9	1.192 (3)	C10—H10A	0.96
O2—C9	1.327 (3)	C10—H10B	0.96
O2—C10	1.442 (3)	C10—H10C	0.96
O3—C11	1.213 (3)	C11—C12	1.481 (3)
O4—C18	1.217 (3)	C12—C13	1.380 (3)
O5—C19	1.413 (3)	C12—C17	1.391 (3)
O5—H5	0.82	C13—C14	1.380 (4)
O6—C20	1.216 (3)	C13—H13	0.93

O7—C27	1.201 (3)	C14—C15	1.386 (4)
N1—C2	1.470 (3)	C14—H14	0.93
N1—C8	1.474 (3)	C15—C16	1.370 (4)
N1—C5	1.475 (3)	C15—H15	0.93
C2—C3	1.528 (3)	C16—C17	1.388 (3)
C2—C18	1.535 (3)	C16—H16	0.93
C2—C11	1.538 (3)	C17—C18	1.468 (3)
C3—C4	1.555 (3)	C18—O4	1.217 (3)
C3—H3A	0.97	C19—C20	1.537 (3)
C3—H3B	0.97	C19—C27	1.544 (3)
C4—C9	1.518 (3)	C20—O6	1.216 (3)
C4—C19	1.553 (3)	C20—C21	1.470 (3)
C4—C5	1.590 (3)	C21—C26	1.382 (4)
C5—C6	1.541 (3)	C21—C22	1.389 (3)
C5—H5A	0.98	C22—C23	1.371 (4)
C6—C7	1.529 (4)	C22—H22	0.93
C6—H6A	0.97	C23—C24	1.376 (5)
C6—H6B	0.97	C23—H23	0.93
C7—C8	1.508 (4)	C24—C25	1.378 (5)
C7—H7A	0.97	C24—H24	0.93
C7—H7B	0.97	C25—C26	1.379 (4)
C8—H8A	0.97	C25—H25	0.93
C8—H8B	0.97	C26—C27	1.479 (4)
C9—O2—C10	116.08 (19)	H10B—C10—H10C	109.5
C19—O5—H5	109.5	O3—C11—C12	126.4 (2)
C2—N1—C8	118.65 (17)	O3—C11—C2	125.6 (2)
C2—N1—C5	107.91 (15)	C12—C11—C2	107.93 (17)
C8—N1—C5	108.00 (16)	C13—C12—C17	120.5 (2)
N1—C2—C3	107.47 (15)	C13—C12—C11	129.8 (2)
N1—C2—C18	104.38 (16)	C17—C12—C11	109.70 (18)
C3—C2—C18	115.39 (17)	C12—C13—C14	117.8 (2)
N1—C2—C11	111.49 (16)	C12—C13—H13	121.1
C3—C2—C11	115.68 (17)	C14—C13—H13	121.1
C18—C2—C11	101.89 (16)	C13—C14—C15	121.5 (2)
C2—C3—C4	106.92 (17)	C13—C14—H14	119.2
C2—C3—H3A	110.3	C15—C14—H14	119.2
C4—C3—H3A	110.3	C16—C15—C14	121.1 (2)
C2—C3—H3B	110.3	C16—C15—H15	119.5
C4—C3—H3B	110.3	C14—C15—H15	119.5
H3A—C3—H3B	108.6	C15—C16—C17	117.7 (2)
C9—C4—C19	105.97 (16)	C15—C16—H16	121.1
C9—C4—C3	111.66 (17)	C17—C16—H16	121.1
C19—C4—C3	112.47 (17)	C16—C17—C12	121.3 (2)
C9—C4—C5	111.40 (16)	C16—C17—C18	128.9 (2)
C19—C4—C5	111.72 (16)	C12—C17—C18	109.77 (18)
C3—C4—C5	103.77 (15)	O4—C18—C17	126.24 (19)
N1—C5—C6	104.63 (17)	O4—C18—C2	125.06 (19)
N1—C5—C4	107.21 (16)	C17—C18—C2	108.67 (17)
C6—C5—C4	119.49 (17)	O5—C19—C20	105.39 (18)

N1—C5—H5A	108.4	O5—C19—C27	104.98 (19)
C6—C5—H5A	108.4	C20—C19—C27	102.48 (17)
C4—C5—H5A	108.4	O5—C19—C4	113.81 (16)
C7—C6—C5	104.81 (18)	C20—C19—C4	114.18 (18)
C7—C6—H6A	110.8	C27—C19—C4	114.79 (19)
C5—C6—H6A	110.8	O6—C20—C21	126.8 (2)
C7—C6—H6B	110.8	O6—C20—C19	124.8 (2)
C5—C6—H6B	110.8	C21—C20—C19	108.40 (19)
H6A—C6—H6B	108.9	C26—C21—C22	121.1 (2)
C8—C7—C6	103.77 (19)	C26—C21—C20	110.3 (2)
C8—C7—H7A	111.0	C22—C21—C20	128.6 (2)
C6—C7—H7A	111.0	C23—C22—C21	117.4 (3)
C8—C7—H7B	111.0	C23—C22—H22	121.3
C6—C7—H7B	111.0	C21—C22—H22	121.3
H7A—C7—H7B	109.0	C22—C23—C24	121.6 (3)
N1—C8—C7	101.33 (18)	C22—C23—H23	119.2
N1—C8—H8A	111.5	C24—C23—H23	119.2
C7—C8—H8A	111.5	C23—C24—C25	121.1 (3)
N1—C8—H8B	111.5	C23—C24—H24	119.4
C7—C8—H8B	111.5	C25—C24—H24	119.4
H8A—C8—H8B	109.3	C24—C25—C26	117.9 (3)
O1—C9—O2	123.1 (2)	C24—C25—H25	121.0
O1—C9—C4	123.0 (2)	C26—C25—H25	121.0
O2—C9—C4	113.85 (17)	C25—C26—C21	120.8 (2)
O2—C10—H10A	109.5	C25—C26—C27	129.0 (3)
O2—C10—H10B	109.5	C21—C26—C27	110.2 (2)
H10A—C10—H10B	109.5	O7—C27—C26	126.8 (2)
O2—C10—H10C	109.5	O7—C27—C19	125.2 (2)
H10A—C10—H10C	109.5	C26—C27—C19	107.8 (2)
C8—N1—C2—C3	−96.3 (2)	C11—C12—C17—C16	177.7 (2)
C5—N1—C2—C3	26.8 (2)	C13—C12—C17—C18	179.1 (2)
C8—N1—C2—C18	140.68 (18)	C11—C12—C17—C18	−1.5 (2)
C5—N1—C2—C18	−96.18 (17)	C16—C17—C18—O4	−4.9 (4)
C8—N1—C2—C11	31.4 (2)	C12—C17—C18—O4	174.1 (2)
C5—N1—C2—C11	154.57 (16)	C16—C17—C18—C2	173.1 (2)
N1—C2—C3—C4	−18.4 (2)	C12—C17—C18—C2	−7.8 (2)
C18—C2—C3—C4	97.5 (2)	N1—C2—C18—O4	75.0 (3)
C11—C2—C3—C4	−143.72 (17)	C3—C2—C18—O4	−42.6 (3)
C2—C3—C4—C9	123.77 (18)	C11—C2—C18—O4	−168.8 (2)
C2—C3—C4—C19	−117.25 (18)	N1—C2—C18—C17	−102.99 (19)
C2—C3—C4—C5	3.7 (2)	C3—C2—C18—C17	139.32 (18)
C2—N1—C5—C6	−152.10 (17)	C11—C2—C18—C17	13.2 (2)
C8—N1—C5—C6	−22.7 (2)	C9—C4—C19—O5	177.89 (19)
C2—N1—C5—C4	−24.3 (2)	C3—C4—C19—O5	55.6 (2)
C8—N1—C5—C4	105.14 (18)	C5—C4—C19—O5	−60.6 (2)
C9—C4—C5—N1	−108.08 (18)	C9—C4—C19—C20	56.8 (2)
C19—C4—C5—N1	133.60 (17)	C3—C4—C19—C20	−65.4 (2)
C3—C4—C5—N1	12.2 (2)	C5—C4—C19—C20	178.33 (17)
C9—C4—C5—C6	10.5 (3)	C9—C4—C19—C27	−61.1 (2)

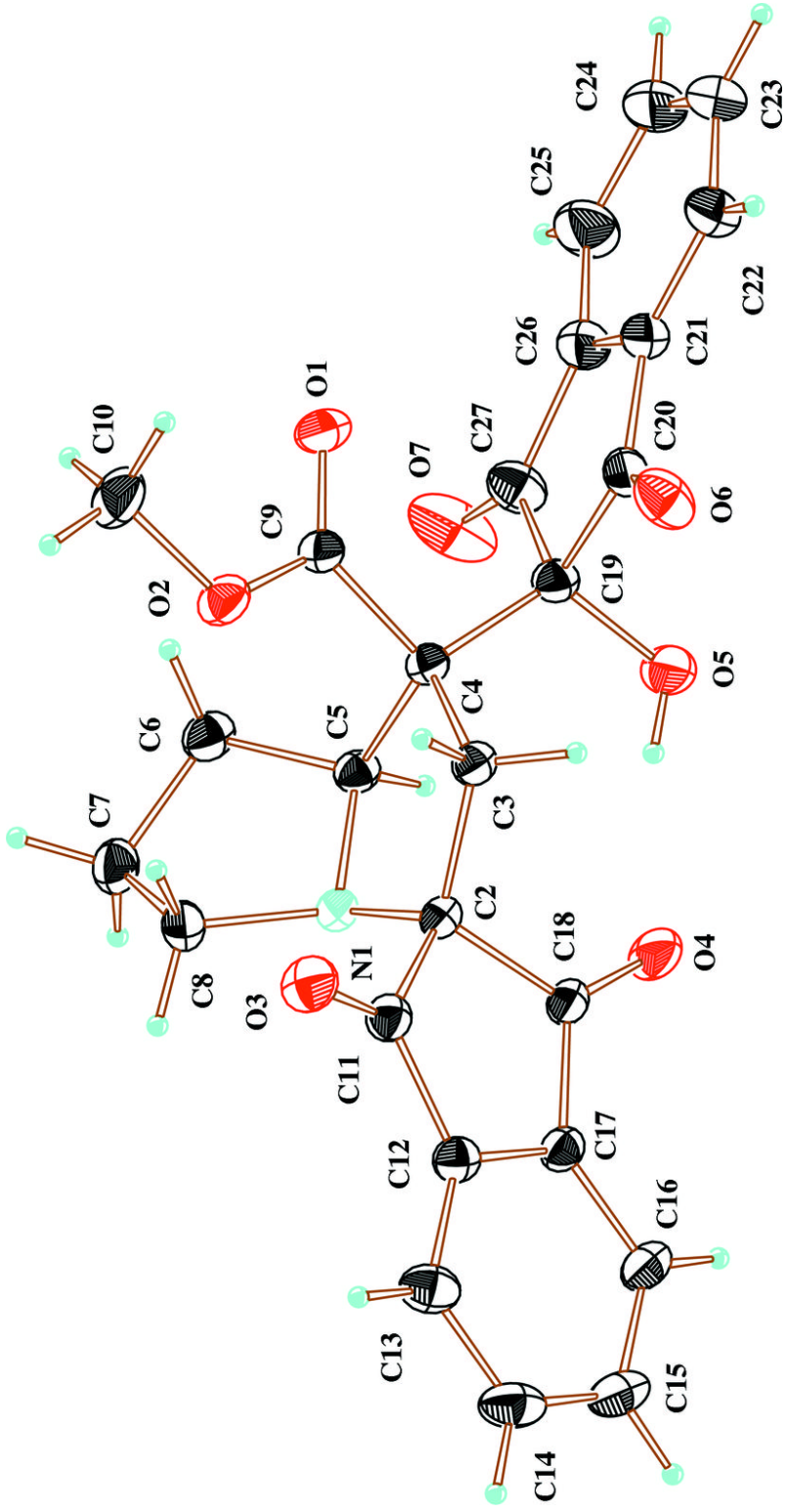
C19—C4—C5—C6	−107.8 (2)	C3—C4—C19—C27	176.63 (18)
C3—C4—C5—C6	130.8 (2)	C5—C4—C19—C27	60.4 (2)
N1—C5—C6—C7	−3.4 (2)	O5—C19—C20—O6	−75.5 (3)
C4—C5—C6—C7	−123.3 (2)	C27—C19—C20—O6	174.9 (2)
C5—C6—C7—C8	27.2 (2)	C4—C19—C20—O6	50.2 (3)
C2—N1—C8—C7	162.84 (18)	O5—C19—C20—C21	101.1 (2)
C5—N1—C8—C7	39.7 (2)	C27—C19—C20—C21	−8.5 (2)
C6—C7—C8—N1	−40.4 (2)	C4—C19—C20—C21	−133.28 (19)
C10—O2—C9—O1	−0.5 (4)	O6—C20—C21—C26	−178.2 (2)
C10—O2—C9—C4	−178.6 (2)	C19—C20—C21—C26	5.3 (3)
C19—C4—C9—O1	18.5 (3)	O6—C20—C21—C22	1.2 (4)
C3—C4—C9—O1	141.3 (2)	C19—C20—C21—C22	−175.3 (2)
C5—C4—C9—O1	−103.2 (3)	C26—C21—C22—C23	−0.5 (4)
C19—C4—C9—O2	−163.39 (18)	C20—C21—C22—C23	−179.8 (3)
C3—C4—C9—O2	−40.6 (2)	C21—C22—C23—C24	0.5 (5)
C5—C4—C9—O2	74.9 (2)	C22—C23—C24—C25	0.1 (6)
N1—C2—C11—O3	−85.2 (3)	C23—C24—C25—C26	−0.8 (5)
C3—C2—C11—O3	37.9 (3)	C24—C25—C26—C21	0.9 (5)
C18—C2—C11—O3	163.9 (2)	C24—C25—C26—C27	179.3 (3)
N1—C2—C11—C12	96.95 (19)	C22—C21—C26—C25	−0.2 (4)
C3—C2—C11—C12	−139.87 (18)	C20—C21—C26—C25	179.2 (3)
C18—C2—C11—C12	−13.9 (2)	C22—C21—C26—C27	−178.9 (2)
O3—C11—C12—C13	11.7 (4)	C20—C21—C26—C27	0.6 (3)
C2—C11—C12—C13	−170.5 (2)	C25—C26—C27—O7	0.0 (5)
O3—C11—C12—C17	−167.6 (2)	C21—C26—C27—O7	178.6 (3)
C2—C11—C12—C17	10.2 (2)	C25—C26—C27—C19	175.3 (3)
C17—C12—C13—C14	0.8 (4)	C21—C26—C27—C19	−6.2 (3)
C11—C12—C13—C14	−178.5 (2)	O5—C19—C27—O7	74.2 (4)
C12—C13—C14—C15	0.1 (4)	C20—C19—C27—O7	−175.9 (3)
C13—C14—C15—C16	−0.2 (4)	C4—C19—C27—O7	−51.5 (4)
C14—C15—C16—C17	−0.7 (4)	O5—C19—C27—C26	−101.1 (2)
C15—C16—C17—C12	1.7 (3)	C20—C19—C27—C26	8.8 (3)
C15—C16—C17—C18	−179.4 (2)	C4—C19—C27—C26	133.2 (2)
C13—C12—C17—C16	−1.8 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O4	0.82	1.89	2.691 (2)	165
C8—H8A···O4^{i}	0.97	2.56	3.259 (3)	130
C16—H16···O3^{ii}	0.93	2.57	3.481 (3)	168
C22—H22···O7^{iii}	0.93	2.38	3.280 (4)	163
C23—H23···O1^{iv}	0.93	2.47	3.238 (3)	140

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

Fig. 1


C24
C23
C25
C22
C26
C21
O1
C10
C27
C20
O7
O6
C9
C19
O2
O5
C4
C6
C5
C3
C7
C2
C18
O4
C8
N1
O3
C11
C12
C17
C16
C13
C15
C14