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10,10a-Dihydroxy-7-methoxy-2,2-dimethyl-5-(2-methylprop-1-en-1-yl)-1,10,10a,14,14a,15b-hexahydro-5*H*,12*H*-3,4-dioxa-5a,11a,15a-triazacyclooct[*lm*]indeno[5,6-*b*]fluorene-11,15(2*H*,13*H*)-dione

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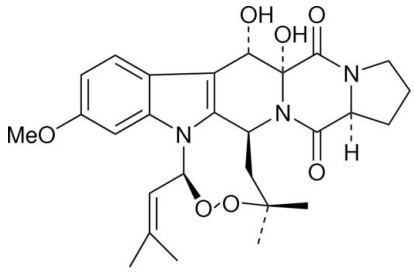
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.102; data-to-parameter ratio = 10.3.

The title compound, $C_{27}H_{33}N_3O_7$, isolated from the marine-derived fungus *Aspergillus sydowi* and also known as verruculogen, contains six fused rings. The indole portion of the molecule adopts a planar conformation with a maximum deviation of 0.022 (2) Å. The six-membered ring with one N atom has a half-chair conformation, the six-membered ring with two N atoms has a boat conformation and the terminal five-membered ring has an envelope conformation. There is an intramolecular hydrogen bond between a hydroxyl group and a carbonyl O atom. Further O—H···O hydrogen bonds link the molecules, forming a one-dimensional chain extending along the *a* axis.

Related literature

For general background, see: Bunger *et al.* (2004); Cockrum *et al.* (1979); Cole *et al.* (1972); Cui *et al.* (1996); Dowd *et al.* (1988); Geris *et al.* (2002); Selala *et al.* (1991); Cremer & Pople (1975).



Experimental

Crystal data

$C_{27}H_{33}N_3O_7$
M_r = 511.56
Orthorhombic, $P2_12_12_1$
a = 11.0919 (15) Å
b = 13.587 (2) Å
c = 17.329 (3) Å
V = 2611.6 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.43 × 0.40 × 0.18 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.96, T_{max} = 0.98
14798 measured reflections
3475 independent reflections
2098 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.102
S = 1.05
3475 reflections
337 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O4^{i}	0.82	1.97	2.676 (3)	144
O6—H6···O4	0.82	2.13	2.747 (4)	132

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2129).

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supplementary materials

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10,10a-Dihydroxy-7-methoxy-2,2-dimethyl-5-(2-methylprop-1-en-1-yl)-1,10,10a,14,14a,15b-hexahydro-5*H*,12*H*-3,4-dioxa-5a,11a,15a-triazacyclooct[*lm*]indeno[5,6-*b*]fluorene-11,15(2*H*,13*H*)-dione

Y.-C. Fang, W.-L. Wang, W. Sun, Q.-Q. Gu and W.-M. Zhu

Comment

TR-2, $C_{27}H_{33}O_7$, (I), was first isolated from the metabolites of *Penicillium verruculosum* (Cole *et al.*, 1972). This title compound (and related ones) arise from their biological activities (Cockrum *et al.*, 1979; Dowd *et al.*, 1988; Selala *et al.*, 1991; Cui *et al.*, 1996; Geris *et al.*, 2002; Bunger *et al.*, 2004). We isolated (I) as a part of our ongoing study characterizing bioactive metabolites from various marine derived microorganism. This paper reports the X-ray crystallographic study of (I).

As shown in Fig. 1, there is an intramolecular hydrogen bond between a hydroxyl (O6) and carbonyl oxygen atom (O4). The indole portion (N1, C1, C11 to C17) of the molecule adopts a planar conformation with the maximum deviation being 0.022 (2) Å (for atom C1). The six-membered N2—C2—C1—C11—C10—C9 ring takes on a half-chair conformation, the six-membered N2—C3—C4—N3—C8—C9 ring adopts a boat conformation, and the five-membered N3—C4—C5—C6—C7 ring takes on an envelope conformation. The corresponding puckering parameters (Cremer & Pople, 1975) are Q = 0.479 (3) Å, θ = 70.1 (4)°, φ = 270.8 (4)°; Q = 0.484 (4) Å, θ = 99.3 (4)°, φ = 124.9 (4)° and Q = 0.367 (4) Å, φ = 257.2 (6)°. The hydroxyls on atom C9 and C10 are *cis*-conformation. The –C(H)═CMe_2 side arm is approximately perpendicular to this plane, as seen in the values of the C17—N1—C18—C19 and C1—N1—C18—C19 torsion angles of 77.1 (3) and −92.3 (3))°, respectively.

In the crystal structure, the molecules interact with each other *via* O—H···O hydrogen bonds (Table 2) to form a 1-D chain along the *a* axis (Fig. 2).

Experimental

The isolated fugal strain *Aspergillus sydowi* PFW-13, was isolated from drift wood collected on the beach of Baishamen, Hainan, China. The working strain was cultured under static conditions at 303 K for 40 days in one hundred and fifty 1000-ml conical flasks containing the liquid medium (300 ml/flask) composed of maltose (20 g/*L*), mannitol (20 g/*L*), glucose (10 g/*L*), monosodium glutamate (10 g/*L*), KH_2PO_4 (5 g/*L*), $MgSO_4$ (0.3 g/*L*), yeast extract (3 g/*L*), corn steep liquor (1 g/*L*) and sea-water after adjusting its pH to 7.0. The fermented whole broth (45 liters) was filtered through cheese cloth to separate into supernatant and mycelia. The former was concentrated under reduced pressure to about a quarter of the original volume and then extracted three times with ethyl acetate to give an ethyl acetate solution, while the latter was extracted three times with acetone. The acetone solution was concentrated under reduced pressure to afford an aqueous solution, which was extracted three times with ethyl acetate to give a second ethyl acetate solution. Both ethyl acetate solutions were combined and concentrated under reduced pressure to give a crude extract (86.7 g) which was subjected to chromatography over silica gel column using a stepwise gradient elution of CHCl3-petroleum ether (0–100%) and then MeOH-CHCl3 (0–50%), to yield eighteen fractions (Fr.1-Fr.18). The fraction 11, eluted with chloroform: methanol 99:1, was further purified into 12 subfractions (Fr.11–1-Fr.11–12) by another silica gel column using a step gradient elution of petroleum ether:acetone. The

title compound (9 mg) was purified by extensive preparative HPLC using MeOH-H_2O from Fr.11–3. The single crystals were obtained by slow evaporation of $CHCl_3$—MeOH (1:1) solution at 299 K.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of 0.93–0.98 (C—H) and 0.82 Å (O—H), and with U_{iso}(H) values of $1.2U_{eq}$(C) and $1.5U_{eq}$(C_{methyl}, O). As mentioned above, the absolute configuration could not be determined crystallographically and Friedel pairs were merged.

Figures

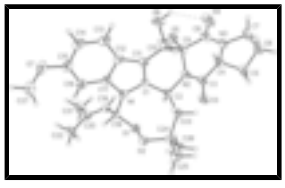


Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids and atom-numbering for non-H atoms. Dashed line indicates hydrogen bond.

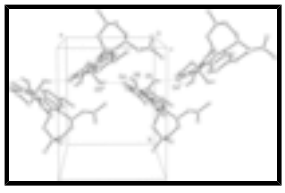


Fig. 2. A view showing the ribbon extending along [100], formed by O—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes:(i) $x - 1/2, 1/2 - y, 2 - z$; (ii) $x + 1/2, 1/2 - y, 2 - z$]

10,10*a*-dihydroxy-7-methoxy-2,2-dimethyl-5-(2-methylprop-1-en-1-yl)- 1,10,10*a*,14,14*a*,15*b*-hexahydro-5H,12*H*-3,4-dioxa-5a,11*a*,15*a*- triazacyclooct[lm]indeno[5,6-*b*]fluorene-11,15(2*H*,13*H*)-dione

Crystal data

$C_{27}H_{33}N_3O_7$	F_{000} = 1088
M_r = 511.56	D_x = 1.301 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 2013 reflections
a = 11.0919 (15) Å	θ = 2.2–22.6º
b = 13.587 (2) Å	μ = 0.10 mm^{-1}
c = 17.329 (3) Å	T = 298 (2) K
V = 2611.6 (7) Å^3	Block, colorless
Z = 4	0.43 × 0.40 × 0.18 mm

Data collection

Bruker APEX area-detector diffractometer	3475 independent reflections
Radiation source: fine-focus sealed tube	2098 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.040
T = 298(2) K	θ_{max} = 28.0º
phi and ω scans	θ_{min} = 1.9º

Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
T_{min} = 0.96, T_{max} = 0.98
14798 measured reflections
h = −14→14
k = −13→17
l = −16→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.102
S = 1.05
3475 reflections
337 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
$w = 1/[\sigma^2(F_o^2) + (0.040P)^2 + 0.3529P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: none

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}
N1	−0.0080 (2)	0.49991 (17)	0.87268 (12)	0.0406 (5)
N2	0.1406 (2)	0.40911 (17)	1.05444 (12)	0.0408 (6)
N3	0.2856 (2)	0.29029 (18)	1.14384 (16)	0.0510 (7)
O1	−0.11729 (18)	0.60443 (14)	0.95499 (10)	0.0485 (5)
O2	−0.0234 (2)	0.68146 (14)	0.95131 (11)	0.0552 (6)
O3	0.0500 (2)	0.46740 (17)	1.16292 (12)	0.0657 (7)
O4	0.3954 (2)	0.2560 (2)	1.03899 (14)	0.0863 (8)
O5	0.1201 (2)	0.24215 (15)	1.01719 (13)	0.0580 (6)
H5	0.0694	0.2498	0.9833	0.087*
O6	0.2858 (3)	0.26652 (19)	0.89711 (14)	0.0849 (9)
H6	0.3307	0.2340	0.9248	0.127*
O7	−0.0077 (3)	0.4553 (2)	0.59212 (13)	0.0854 (8)

C1	0.0662 (2)	0.4571 (2)	0.92763 (15)	0.0378 (6)
C2	0.0680 (2)	0.48387 (19)	1.01124 (15)	0.0391 (6)
H2	−0.0146	0.4847	1.0313	0.047*
C3	0.1168 (3)	0.4079 (2)	1.13172 (16)	0.0466 (7)
C4	0.1758 (3)	0.3289 (2)	1.17920 (17)	0.0510 (8)
H4	0.1185	0.2746	1.1854	0.061*
C5	0.2188 (3)	0.3601 (3)	1.25855 (19)	0.0649 (10)
H5A	0.1534	0.3586	1.2957	0.078*
H5B	0.2531	0.4258	1.2573	0.078*
C6	0.3142 (4)	0.2834 (3)	1.2777 (2)	0.0751 (11)
H6A	0.3707	0.3085	1.3156	0.090*
H6B	0.2774	0.2239	1.2976	0.090*
C7	0.3765 (3)	0.2638 (3)	1.2018 (2)	0.0707 (10)
H7A	0.4481	0.3042	1.1964	0.085*
H7B	0.3991	0.1951	1.1972	0.085*
C8	0.3026 (3)	0.2881 (2)	1.0690 (2)	0.0532 (8)
C9	0.1980 (2)	0.3232 (2)	1.01831 (17)	0.0424 (7)
C10	0.2457 (3)	0.3501 (2)	0.93781 (17)	0.0507 (8)
H10	0.3132	0.3960	0.9435	0.061*
C11	0.1487 (2)	0.3989 (2)	0.89247 (16)	0.0420 (7)
C12	0.1258 (3)	0.4018 (2)	0.81131 (15)	0.0439 (7)
C13	0.1757 (3)	0.3572 (2)	0.74574 (18)	0.0573 (9)
H13	0.2414	0.3151	0.7502	0.069*
C14	0.1261 (4)	0.3768 (2)	0.67513 (19)	0.0644 (10)
H14	0.1578	0.3460	0.6317	0.077*
C15	0.0300 (3)	0.4410 (2)	0.66623 (17)	0.0581 (9)
C16	−0.0222 (3)	0.4859 (2)	0.72910 (16)	0.0517 (8)
H16	−0.0880	0.5278	0.7239	0.062*
C17	0.0283 (3)	0.4654 (2)	0.80115 (15)	0.0407 (7)
C18	−0.1171 (3)	0.5578 (2)	0.88253 (15)	0.0417 (7)
H18	−0.1213	0.6078	0.8419	0.050*
C19	−0.2284 (3)	0.4966 (2)	0.88037 (16)	0.0443 (7)
H19	−0.2297	0.4406	0.9112	0.053*
C20	−0.3257 (3)	0.5148 (2)	0.83855 (17)	0.0488 (7)
C21	−0.4331 (3)	0.4488 (3)	0.8417 (2)	0.0767 (11)
H21A	−0.4195	0.3977	0.8788	0.115*
H21B	−0.4463	0.4201	0.7918	0.115*
H21C	−0.5027	0.4864	0.8564	0.115*
C22	−0.3384 (4)	0.6008 (3)	0.7849 (2)	0.0765 (11)
H22A	−0.4123	0.6348	0.7958	0.115*
H22B	−0.3395	0.5777	0.7325	0.115*
H22C	−0.2716	0.6448	0.7920	0.115*
C23	0.1265 (3)	0.5860 (2)	1.02369 (17)	0.0459 (7)
H23A	0.1676	0.5847	1.0731	0.055*
H23B	0.1878	0.5946	0.9843	0.055*
C24	0.0459 (3)	0.6763 (2)	1.02237 (17)	0.0502 (7)
C25	0.1227 (4)	0.7684 (2)	1.0157 (2)	0.0713 (10)
H25A	0.1679	0.7663	0.9685	0.107*
H25B	0.1772	0.7718	1.0586	0.107*

H25C	0.0716	0.8254	1.0157	0.107*
C26	−0.0372 (3)	0.6817 (3)	1.09160 (18)	0.0627 (9)
H26A	−0.0913	0.7363	1.0857	0.094*
H26B	0.0098	0.6903	1.1376	0.094*
H26C	−0.0828	0.6218	1.0954	0.094*
C27	−0.1107 (5)	0.5151 (4)	0.5801 (2)	0.1091 (17)
H27A	−0.1277	0.5191	0.5259	0.164*
H27B	−0.0957	0.5799	0.5999	0.164*
H27C	−0.1786	0.4869	0.6064	0.164*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0425 (13)	0.0406 (13)	0.0386 (13)	0.0066 (12)	−0.0005 (11)	−0.0020 (11)
N2	0.0357 (13)	0.0410 (13)	0.0457 (14)	0.0082 (11)	0.0021 (10)	0.0047 (10)
N3	0.0455 (15)	0.0510 (15)	0.0567 (17)	0.0080 (12)	−0.0114 (13)	0.0074 (12)
O1	0.0420 (11)	0.0484 (11)	0.0551 (12)	0.0040 (10)	−0.0061 (10)	−0.0114 (9)
O2	0.0661 (14)	0.0395 (11)	0.0601 (13)	−0.0002 (11)	−0.0174 (11)	−0.0020 (10)
O3	0.0705 (16)	0.0773 (17)	0.0494 (13)	0.0286 (14)	0.0093 (12)	0.0060 (11)
O4	0.0528 (15)	0.113 (2)	0.0930 (19)	0.0406 (16)	0.0072 (14)	0.0165 (17)
O5	0.0568 (14)	0.0445 (11)	0.0727 (16)	−0.0053 (11)	−0.0104 (12)	0.0062 (11)
O6	0.098 (2)	0.0834 (18)	0.0737 (16)	0.0550 (16)	0.0134 (15)	−0.0067 (14)
O7	0.119 (2)	0.094 (2)	0.0431 (14)	0.0029 (19)	−0.0075 (15)	−0.0063 (13)
C1	0.0361 (15)	0.0347 (14)	0.0427 (15)	0.0023 (12)	0.0018 (13)	0.0019 (12)
C2	0.0321 (14)	0.0389 (15)	0.0462 (16)	0.0063 (12)	−0.0005 (12)	0.0036 (13)
C3	0.0429 (17)	0.0500 (17)	0.0470 (17)	0.0047 (17)	0.0040 (14)	0.0058 (15)
C4	0.0460 (18)	0.0472 (18)	0.0599 (19)	−0.0008 (16)	−0.0030 (16)	0.0120 (15)
C5	0.075 (2)	0.062 (2)	0.058 (2)	0.005 (2)	−0.0133 (18)	0.0070 (17)
C6	0.082 (3)	0.075 (3)	0.068 (2)	0.005 (2)	−0.024 (2)	0.0159 (19)
C7	0.061 (2)	0.063 (2)	0.089 (3)	0.008 (2)	−0.030 (2)	0.0166 (19)
C8	0.0395 (18)	0.0466 (18)	0.074 (2)	0.0093 (15)	−0.0034 (17)	0.0096 (16)
C9	0.0331 (15)	0.0366 (15)	0.0576 (18)	0.0039 (13)	0.0031 (14)	0.0024 (14)
C10	0.0411 (17)	0.0507 (18)	0.0604 (19)	0.0113 (15)	0.0089 (15)	0.0019 (15)
C11	0.0379 (17)	0.0406 (15)	0.0476 (17)	0.0033 (14)	0.0060 (13)	0.0032 (13)
C12	0.0445 (17)	0.0418 (16)	0.0452 (16)	−0.0043 (16)	0.0106 (14)	−0.0023 (13)
C13	0.061 (2)	0.056 (2)	0.055 (2)	−0.0004 (18)	0.0195 (17)	−0.0071 (16)
C14	0.078 (3)	0.062 (2)	0.053 (2)	−0.005 (2)	0.021 (2)	−0.0120 (16)
C15	0.079 (2)	0.056 (2)	0.0400 (18)	−0.015 (2)	0.0038 (17)	−0.0049 (15)
C16	0.060 (2)	0.0460 (18)	0.0488 (18)	−0.0035 (17)	−0.0027 (16)	−0.0024 (14)
C17	0.0439 (17)	0.0385 (15)	0.0398 (16)	−0.0064 (14)	0.0023 (13)	−0.0018 (13)
C18	0.0443 (16)	0.0439 (16)	0.0369 (15)	0.0074 (15)	−0.0046 (13)	−0.0044 (12)
C19	0.0455 (18)	0.0399 (15)	0.0474 (17)	0.0016 (15)	0.0013 (14)	−0.0002 (13)
C20	0.0456 (17)	0.0457 (18)	0.0550 (18)	0.0002 (15)	−0.0088 (15)	−0.0021 (15)
C21	0.060 (2)	0.072 (2)	0.098 (3)	−0.012 (2)	−0.019 (2)	0.009 (2)
C22	0.069 (3)	0.069 (2)	0.092 (3)	−0.004 (2)	−0.030 (2)	0.018 (2)
C23	0.0427 (16)	0.0473 (16)	0.0476 (16)	0.0005 (14)	−0.0050 (15)	0.0018 (14)
C24	0.0556 (19)	0.0411 (16)	0.0539 (18)	0.0013 (15)	−0.0128 (16)	−0.0047 (15)
C25	0.084 (3)	0.0454 (19)	0.085 (3)	−0.0064 (19)	−0.020 (2)	−0.0006 (18)

C26	0.068 (2)	0.059 (2)	0.061 (2)	0.0131 (19)	−0.0027 (18)	−0.0170 (17)
C27	0.168 (5)	0.102 (4)	0.057 (2)	0.018 (4)	−0.035 (3)	−0.001 (2)

Geometric parameters (Å, °)

N1—C17	1.385 (3)	C10—H10	0.9800
N1—C1	1.386 (3)	C11—C12	1.430 (4)
N1—C18	1.454 (3)	C12—C17	1.396 (4)
N2—C3	1.365 (3)	C12—C13	1.401 (4)
N2—C9	1.469 (3)	C13—C14	1.368 (5)
N2—C2	1.497 (3)	C13—H13	0.9300
N3—C8	1.311 (4)	C14—C15	1.386 (5)
N3—C4	1.460 (4)	C14—H14	0.9300
N3—C7	1.468 (4)	C15—C16	1.376 (4)
O1—C18	1.407 (3)	C16—C17	1.397 (4)
O1—O2	1.478 (3)	C16—H16	0.9300
O2—C24	1.453 (3)	C18—C19	1.488 (4)
O3—C3	1.223 (3)	C18—H18	0.9800
O4—C8	1.233 (4)	C19—C20	1.323 (4)
O5—C9	1.400 (3)	C19—H19	0.9300
O5—H5	0.8200	C20—C21	1.492 (5)
O6—C10	1.409 (4)	C20—C22	1.500 (4)
O6—H6	0.8200	C21—H21A	0.9600
O7—C15	1.364 (4)	C21—H21B	0.9600
O7—C27	1.418 (5)	C21—H21C	0.9600
C1—C11	1.354 (4)	C22—H22A	0.9600
C1—C2	1.494 (4)	C22—H22B	0.9600
C2—C23	1.547 (4)	C22—H22C	0.9600
C2—H2	0.9800	C23—C24	1.519 (4)
C3—C4	1.502 (4)	C23—H23A	0.9700
C4—C5	1.516 (4)	C23—H23B	0.9700
C4—H4	0.9800	C24—C26	1.515 (4)
C5—C6	1.522 (5)	C24—C25	1.519 (4)
C5—H5A	0.9700	C25—H25A	0.9600
C5—H5B	0.9700	C25—H25B	0.9600
C6—C7	1.510 (5)	C25—H25C	0.9600
C6—H6A	0.9700	C26—H26A	0.9600
C6—H6B	0.9700	C26—H26B	0.9600
C7—H7A	0.9700	C26—H26C	0.9600
C7—H7B	0.9700	C27—H27A	0.9600
C8—C9	1.532 (4)	C27—H27B	0.9600
C9—C10	1.536 (4)	C27—H27C	0.9600
C10—C11	1.488 (4)		
C17—N1—C1	107.5 (2)	C13—C12—C11	135.7 (3)
C17—N1—C18	122.0 (2)	C14—C13—C12	118.9 (3)
C1—N1—C18	129.8 (2)	C14—C13—H13	120.6
C3—N2—C9	119.5 (2)	C12—C13—H13	120.6
C3—N2—C2	113.3 (2)	C13—C14—C15	122.1 (3)
C9—N2—C2	124.0 (2)	C13—C14—H14	119.0

C8—N3—C4	122.9 (3)	C15—C14—H14	119.0
C8—N3—C7	124.9 (3)	O7—C15—C16	123.6 (3)
C4—N3—C7	112.0 (3)	O7—C15—C14	115.4 (3)
C18—O1—O2	106.2 (2)	C16—C15—C14	121.0 (3)
C24—O2—O1	107.57 (19)	C15—C16—C17	116.8 (3)
C9—O5—H5	109.5	C15—C16—H16	121.6
C10—O6—H6	109.5	C17—C16—H16	121.6
C15—O7—C27	117.8 (3)	N1—C17—C12	108.8 (2)
C11—C1—N1	109.7 (2)	N1—C17—C16	128.0 (3)
C11—C1—C2	124.7 (3)	C12—C17—C16	123.2 (3)
N1—C1—C2	124.9 (2)	O1—C18—N1	110.5 (2)
C1—C2—N2	109.1 (2)	O1—C18—C19	105.8 (2)
C1—C2—C23	111.1 (2)	N1—C18—C19	112.7 (2)
N2—C2—C23	108.3 (2)	O1—C18—H18	109.3
C1—C2—H2	109.5	N1—C18—H18	109.3
N2—C2—H2	109.5	C19—C18—H18	109.2
C23—C2—H2	109.5	C20—C19—C18	125.9 (3)
O3—C3—N2	122.9 (3)	C20—C19—H19	117.1
O3—C3—C4	119.6 (3)	C18—C19—H19	117.1
N2—C3—C4	117.5 (3)	C19—C20—C21	121.3 (3)
N3—C4—C3	113.0 (2)	C19—C20—C22	124.2 (3)
N3—C4—C5	102.7 (3)	C21—C20—C22	114.5 (3)
C3—C4—C5	115.7 (3)	C20—C21—H21A	109.5
N3—C4—H4	108.4	C20—C21—H21B	109.5
C3—C4—H4	108.4	H21A—C21—H21B	109.5
C5—C4—H4	108.4	C20—C21—H21C	109.5
C4—C5—C6	103.0 (3)	H21A—C21—H21C	109.5
C4—C5—H5A	111.2	H21B—C21—H21C	109.5
C6—C5—H5A	111.2	C20—C22—H22A	109.5
C4—C5—H5B	111.2	C20—C22—H22B	109.5
C6—C5—H5B	111.2	H22A—C22—H22B	109.5
H5A—C5—H5B	109.1	C20—C22—H22C	109.5
C7—C6—C5	104.4 (3)	H22A—C22—H22C	109.5
C7—C6—H6A	110.9	H22B—C22—H22C	109.5
C5—C6—H6A	110.9	C24—C23—C2	118.4 (2)
C7—C6—H6B	110.9	C24—C23—H23A	107.7
C5—C6—H6B	110.9	C2—C23—H23A	107.7
H6A—C6—H6B	108.9	C24—C23—H23B	107.7
N3—C7—C6	103.8 (3)	C2—C23—H23B	107.7
N3—C7—H7A	111.0	H23A—C23—H23B	107.1
C6—C7—H7A	111.0	O2—C24—C26	110.3 (2)
N3—C7—H7B	111.0	O2—C24—C25	101.1 (2)
C6—C7—H7B	111.0	C26—C24—C25	111.2 (3)
H7A—C7—H7B	109.0	O2—C24—C23	111.3 (2)
O4—C8—N3	123.1 (3)	C26—C24—C23	112.7 (3)
O4—C8—C9	120.0 (3)	C25—C24—C23	109.6 (3)
N3—C8—C9	116.8 (3)	C24—C25—H25A	109.5
O5—C9—N2	111.3 (2)	C24—C25—H25B	109.5
O5—C9—C8	103.3 (2)	H25A—C25—H25B	109.5

N2—C9—C8	109.4 (2)	C24—C25—H25C	109.5
O5—C9—C10	112.8 (3)	H25A—C25—H25C	109.5
N2—C9—C10	110.3 (2)	H25B—C25—H25C	109.5
C8—C9—C10	109.5 (2)	C24—C26—H26A	109.5
O6—C10—C11	108.9 (3)	C24—C26—H26B	109.5
O6—C10—C9	111.8 (2)	H26A—C26—H26B	109.5
C11—C10—C9	109.7 (2)	C24—C26—H26C	109.5
O6—C10—H10	108.8	H26A—C26—H26C	109.5
C11—C10—H10	108.8	H26B—C26—H26C	109.5
C9—C10—H10	108.8	O7—C27—H27A	109.5
C1—C11—C12	107.9 (3)	O7—C27—H27B	109.5
C1—C11—C10	120.7 (3)	H27A—C27—H27B	109.5
C12—C11—C10	131.3 (3)	O7—C27—H27C	109.5
C17—C12—C13	118.1 (3)	H27A—C27—H27C	109.5
C17—C12—C11	106.2 (2)	H27B—C27—H27C	109.5
C18—O1—O2—C24	−135.2 (2)	N2—C9—C10—C11	−50.7 (3)
C17—N1—C1—C11	1.3 (3)	C8—C9—C10—C11	−171.1 (2)
C18—N1—C1—C11	171.8 (3)	N1—C1—C11—C12	−1.5 (3)
C17—N1—C1—C2	171.9 (3)	C2—C1—C11—C12	−172.2 (3)
C18—N1—C1—C2	−17.5 (4)	N1—C1—C11—C10	175.2 (2)
C11—C1—C2—N2	−21.9 (4)	C2—C1—C11—C10	4.6 (4)
N1—C1—C2—N2	168.8 (2)	O6—C10—C11—C1	156.1 (3)
C11—C1—C2—C23	97.3 (3)	C9—C10—C11—C1	33.5 (4)
N1—C1—C2—C23	−71.9 (3)	O6—C10—C11—C12	−28.0 (4)
C3—N2—C2—C1	−160.2 (2)	C9—C10—C11—C12	−150.6 (3)
C9—N2—C2—C1	−0.8 (3)	C1—C11—C12—C17	1.1 (3)
C3—N2—C2—C23	78.9 (3)	C10—C11—C12—C17	−175.2 (3)
C9—N2—C2—C23	−121.8 (3)	C1—C11—C12—C13	−178.2 (3)
C9—N2—C3—O3	−165.9 (3)	C10—C11—C12—C13	5.5 (6)
C2—N2—C3—O3	−5.5 (4)	C17—C12—C13—C14	−0.8 (4)
C9—N2—C3—C4	13.8 (4)	C11—C12—C13—C14	178.4 (3)
C2—N2—C3—C4	174.2 (2)	C12—C13—C14—C15	1.6 (5)
C8—N3—C4—C3	−29.2 (4)	C27—O7—C15—C16	−3.8 (5)
C7—N3—C4—C3	145.4 (3)	C27—O7—C15—C14	175.9 (3)
C8—N3—C4—C5	−154.5 (3)	C13—C14—C15—O7	178.3 (3)
C7—N3—C4—C5	20.1 (3)	C13—C14—C15—C16	−2.0 (5)
O3—C3—C4—N3	−156.7 (3)	O7—C15—C16—C17	−178.7 (3)
N2—C3—C4—N3	23.6 (4)	C14—C15—C16—C17	1.7 (4)
O3—C3—C4—C5	−38.8 (4)	C1—N1—C17—C12	−0.6 (3)
N2—C3—C4—C5	141.5 (3)	C18—N1—C17—C12	−172.0 (2)
N3—C4—C5—C6	−34.5 (3)	C1—N1—C17—C16	177.9 (3)
C3—C4—C5—C6	−158.0 (3)	C18—N1—C17—C16	6.4 (4)
C4—C5—C6—C7	37.2 (4)	C13—C12—C17—N1	179.1 (3)
C8—N3—C7—C6	177.4 (3)	C11—C12—C17—N1	−0.3 (3)
C4—N3—C7—C6	2.9 (4)	C13—C12—C17—C16	0.6 (4)
C5—C6—C7—N3	−24.8 (4)	C11—C12—C17—C16	−178.9 (3)
C4—N3—C8—O4	179.6 (3)	C15—C16—C17—N1	−179.2 (3)
C7—N3—C8—O4	5.7 (5)	C15—C16—C17—C12	−1.0 (4)
C4—N3—C8—C9	−3.3 (4)	O2—O1—C18—N1	70.0 (2)

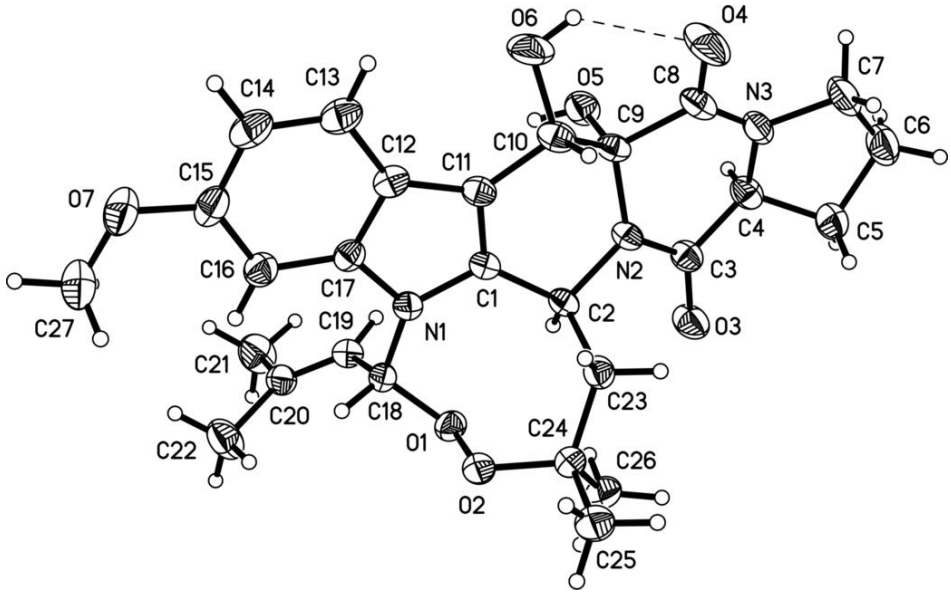
C7—N3—C8—C9	−177.2 (3)	O2—O1—C18—C19	−167.69 (19)
C3—N2—C9—O5	68.8 (3)	C17—N1—C18—O1	−164.8 (2)
C2—N2—C9—O5	−89.4 (3)	C1—N1—C18—O1	25.9 (4)
C3—N2—C9—C8	−44.7 (3)	C17—N1—C18—C19	77.1 (3)
C2—N2—C9—C8	157.1 (2)	C1—N1—C18—C19	−92.3 (3)
C3—N2—C9—C10	−165.3 (3)	O1—C18—C19—C20	108.2 (3)
C2—N2—C9—C10	36.5 (3)	N1—C18—C19—C20	−130.9 (3)
O4—C8—C9—O5	97.9 (3)	C18—C19—C20—C21	−179.2 (3)
N3—C8—C9—O5	−79.3 (3)	C18—C19—C20—C22	0.9 (5)
O4—C8—C9—N2	−143.5 (3)	C1—C2—C23—C24	90.2 (3)
N3—C8—C9—N2	39.3 (3)	N2—C2—C23—C24	−150.0 (2)
O4—C8—C9—C10	−22.5 (4)	O1—O2—C24—C26	−53.3 (3)
N3—C8—C9—C10	160.3 (3)	O1—O2—C24—C25	−171.1 (2)
O5—C9—C10—O6	−46.4 (3)	O1—O2—C24—C23	72.5 (3)
N2—C9—C10—O6	−171.5 (2)	C2—C23—C24—O2	−55.1 (3)
C8—C9—C10—O6	68.0 (3)	C2—C23—C24—C26	69.5 (3)
O5—C9—C10—C11	74.4 (3)	C2—C23—C24—C25	−166.1 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O4^{i}	0.82	1.97	2.676 (3)	144
O6—H6···O4	0.82	2.13	2.747 (4)	132

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

Fig. 1


O6
O4
C14
C13
O5
C8
C7
N3
C9
C6
C12
C10
C11
C15
O7
C4
C5
C16
C17
N2
C3
C1
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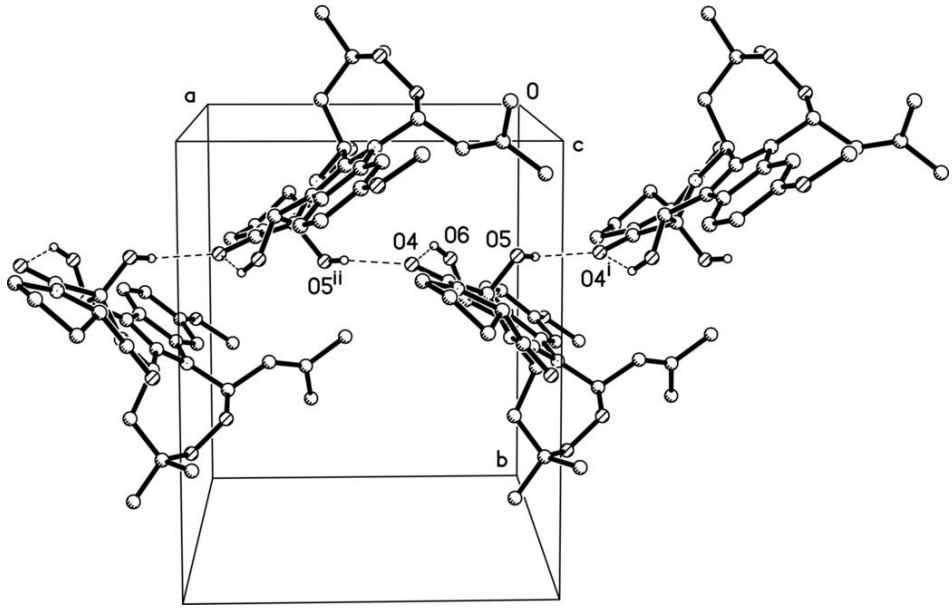
Fig. 2