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(3a*S*,5a*R*,6*R*,8a*R*)-3a-Hydroxy-5a-methyl-6-[(1*R*,2*E*,4*R*)-1,4,5-trimethyl-2-hexen-1-yl]-3a,4,5,5a,6,7,8,8a-octahydro-2*H*-cyclopenta[e]benzofuran-2-one

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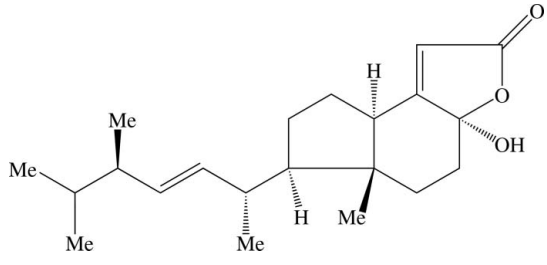
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.045; wR factor = 0.113; data-to-parameter ratio = 8.8.

The title compound, $C_{21}H_{32}O_3$, also known as dimethylincisterol A3, was isolated from halotolerant fungus THW-18. It is composed of three fused rings and a side chain. In the crystal structure, the molecules interact with each other *via* O—H···O hydrogen bonds, resulting in an extended chain along the *b* axis. The absolute configuration was assigned from the measured optical rotation and reference to the literature.

Related literature

For general background, see: Mansoor *et al.* (2005); Togashi *et al.* (1998); Kawagishi *et al.* (2006); De Riccardis *et al.* (1995). For related literature, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{21}H_{32}O_3$
M_r = 332.47
Monoclinic, $P2_1$
a = 8.0506 (9) Å
b = 6.7640 (8) Å
c = 18.858 (2) Å
β = 95.639 (2)°
V = 1021.9 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 298 (2) K
0.56 × 0.51 × 0.18 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.962, T_{max} = 0.988
5311 measured reflections
1965 independent reflections
1247 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.113
S = 1.05
1965 reflections
224 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^{i}	0.82	1.96	2.771 (4)	171

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

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: AV3110).

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supplementary materials

Acta Cryst. (2007). E**63**, o4196 [**doi:10.1107/S1600536807047307**]

(3a*S*,5a*R*,6*R*,8a*R*)-3a-Hydroxy-5a-methyl-6-[(1*R*,2*E*,4*R*)-1,4,5-trimethyl-2-hexen-1-yl]-3a,4,5,5a,6,7,8,8a-octahydro-2*H*-cyclopenta[*e*]benzofuran-2-one

W. Wang, H. Tao, W. Sun, Q.-Q. Gu and W.-M. Zhu

Comment

Dimethylincisterol A3, (I), was first reported as an intermediate to synthesize the (17*R*)-17-methylincisterol (De Riccardis *et al.*, 1995), and then was isolated from a marine sponge *Homaxinella sp.* in 2005 (Mansoor *et al.*, 2005). This title compound and related compounds are of interest because of their biological activities (Togashi *et al.*, 1998; Mansoor *et al.*, 2005; Kawagishi *et al.*, 2006). We isolated (I) as a part of our ongoing study characterizing bioactive metabolites from various halotolerant microorganisms. This is our first report about the title compound isolated from halotolerant fungus THW-18 and the results of its X-ray crystallographic study (Fig. 1).

The five-membered N3—C4—C5—C6—C7 ring adopts a twist conformation, and the six-membered N2—C3—C4—N3—C8—C9 ring adopts a half-chair conformation. The corresponding puckering parameters (Cremer & Pople, 1975) are Q = 0.462 (4) Å, φ = 192.5 (5)° and Q = 0.564 (4) Å, θ = 6.1 (4)°, φ = 264 (3)°.

It was not possible to determined the absolute configuration of compound (I) by anomalous dispersion effects, but the positive optical rotation suggested that this compound was dimethylincisterol A3 (Mansoor *et al.*, 2005; Kawagishi *et al.*, 2006).

As shown in Fig. 2, the compounds are linked into a 1-D ribbon along [010] by the O3—H3A···O2^{i} hydrogen bond. (symmetry code: i = 3 − *x*, 1/2 + *y*, 1 − *z*).

Experimental

The isolated halotolerant fungal strain THW-18, was isolated from the sediments collected in Hongdao salt field, Qingdao, China. The working strain was cultured under static conditions at 303 K for 45 days in two hundred and fifty 1000-ml conical flasks containing the liquid medium (300 ml/flask) composed of maltose (8 g/*L*), mannitol (8 g/*L*), glucose (4 g/*L*), monosodium glutamate (10 g/*L*), KH_2PO_4 (0.5 g/*L*), yeast extract paste (3 g/*L*), maize paste (1 g/*L*), and sea salt (100 g/*L*) after adjusting its pH to 6.5. The fermented whole broth (75 *L*) was filtered through cheese cloth to separate into supernatant and mycelia. The mycelia were extracted three times with acetone and the acetone solution was concentrated under reduced pressure to afford crude extract (42 g). The crude extract, which was subjected to chromatography over silica gel column using a stepwise gradient elution of petroleum ether-$CHCl_3$—MeOH, to yield five fractions (Fr.1-Fr.5). Fr.2 was subjected to Sephadex LH-20 eluting with $CHCl_3$—MeOH (1:1), followed by chromatographing on a silica gel column eluting with $CHCl_3$—MeOH (30:1) to afford three subfractions (Fr.2–2-1-Fr.2–2-3). The title compound (23 mg) was purified by extensive preparative HPLC using MeOH-H_2O (9:1) from Fr.2–2-2. The single crystals were obtained by slow evaporation of a petroleum ether-acetone (1:1) solution at room temperature on the third day.

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

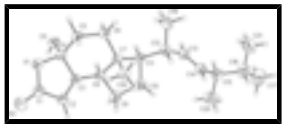

Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

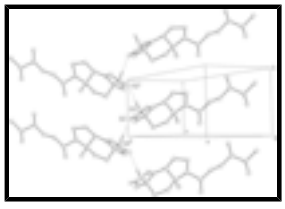

Fig. 2. A view showing the ribbon extending along [010], formed by O—H···O hydrogen bonds (dashed lines). [Symmetry codes: (i) 3 − *x*, 1/2 + *y*, 1 − *z*; (ii) 3 − *x*, −1/2 + *y*, 1 − *z*]

(3aS,5aR,6*R*,8aR)-3a-Hydroxy-5a-methyl-6-[(1*R*,2E,4*R*)-1,4,5-trimethyl-2-hexen- 1-yl]-3a,4,5,5a,6,7,8,8a-octahydro-2*H*-cyclopenta[e]benzofuran-2-one

Crystal data

$C_{21}H_{32}O_3$	F_{000} = 364
M_r = 332.47	D_x = 1.080 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.0506 (9) Å	Cell parameters from 2840 reflections
b = 6.7640 (8) Å	θ = 2.3–22.1º
c = 18.858 (2) Å	μ = 0.07 mm^{-1}
β = 95.639 (2)º	T = 298 (2) K
V = 1021.9 (2) Å^3	Flake, colourless
Z = 2	0.56 × 0.51 × 0.18 mm

Data collection

Bruker APEX area-detector diffractometer	1965 independent reflections
Radiation source: fine-focus sealed tube	1247 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.049
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	h = −9→9
T_{min} = 0.962, T_{max} = 0.988	k = −8→8
5311 measured reflections	l = −22→13

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.045$	$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.113$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.13$ e Å^{-3}
1965 reflections	$\Delta\rho_{min} = -0.14$ e Å^{-3}
224 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack, H. D. (1983). Acta Cryst. A39, 876–881.
Primary atom site location: structure-invariant direct methods	Flack parameter: 0 (10)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2$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	1.4534 (3)	0.2461 (4)	0.46674 (13)	0.0650 (8)
O2	1.4101 (3)	0.2856 (5)	0.58115 (15)	0.0768 (9)
O3	1.4982 (3)	0.4173 (5)	0.36379 (13)	0.0699 (8)
H3	1.5217	0.5222	0.3842	0.105*
C1	1.3731 (4)	0.3295 (6)	0.5192 (2)	0.0601 (10)
C2	1.2432 (4)	0.4584 (6)	0.48848 (19)	0.0558 (10)
H2	1.1721	0.5330	0.5138	0.067*
C3	1.2414 (4)	0.4536 (5)	0.41862 (18)	0.0471 (9)
C4	1.3778 (4)	0.3200 (6)	0.39805 (19)	0.0526 (10)
C5	1.3090 (4)	0.1516 (6)	0.3518 (2)	0.0615 (11)
H5A	1.2516	0.0595	0.3804	0.074*
H5B	1.4002	0.0816	0.3330	0.074*
C6	1.1873 (4)	0.2264 (6)	0.28980 (18)	0.0576 (10)
H6A	1.2492	0.3018	0.2574	0.069*
H6B	1.1380	0.1137	0.2638	0.069*

C7	1.0479 (4)	0.3558 (5)	0.31402 (17)	0.0447 (8)
C8	1.1314 (4)	0.5317 (5)	0.35810 (18)	0.0462 (9)
H8	1.2034	0.5999	0.3270	0.055*
C9	0.9890 (4)	0.6699 (6)	0.36851 (19)	0.0589 (10)
H9A	1.0285	0.8045	0.3760	0.071*
H9B	0.9309	0.6295	0.4088	0.071*
C10	0.8755 (4)	0.6518 (6)	0.29844 (19)	0.0601 (10)
H10A	0.8795	0.7724	0.2709	0.072*
H10B	0.7610	0.6289	0.3082	0.072*
C11	0.9407 (4)	0.4739 (5)	0.25643 (17)	0.0515 (9)
H11	1.0169	0.5279	0.2239	0.062*
C12	0.7992 (4)	0.3695 (6)	0.21038 (19)	0.0614 (11)
H12	0.7251	0.3099	0.2427	0.074*
C13	0.6990 (5)	0.5150 (7)	0.16491 (19)	0.0665 (11)
H13	0.7526	0.5764	0.1294	0.080*
C14	0.5453 (5)	0.5652 (7)	0.1695 (2)	0.0693 (12)
H14	0.4914	0.4964	0.2033	0.083*
C15	0.4421 (5)	0.7175 (7)	0.1283 (2)	0.0797 (14)
H15	0.5070	0.7697	0.0912	0.096*
C16	0.2811 (6)	0.6235 (10)	0.0917 (3)	0.1076 (19)
H16	0.2198	0.5626	0.1285	0.129*
C17	0.1679 (7)	0.7832 (14)	0.0527 (3)	0.172 (3)
H17A	0.2280	0.8488	0.0180	0.258*
H17B	0.1358	0.8781	0.0867	0.258*
H17C	0.0699	0.7216	0.0293	0.258*
C18	0.9365 (4)	0.2377 (6)	0.35926 (19)	0.0560 (10)
H18A	0.9008	0.1185	0.3346	0.084*
H18B	0.8406	0.3154	0.3677	0.084*
H18C	0.9977	0.2047	0.4039	0.084*
C19	0.8610 (6)	0.2045 (7)	0.1650 (2)	0.0952 (16)
H19A	0.7688	0.1507	0.1350	0.143*
H19B	0.9110	0.1024	0.1953	0.143*
H19C	0.9424	0.2563	0.1359	0.143*
C20	0.4074 (8)	0.8880 (10)	0.1784 (3)	0.145 (3)
H20A	0.3362	0.8424	0.2130	0.217*
H20B	0.3535	0.9941	0.1512	0.217*
H20C	0.5108	0.9343	0.2024	0.217*
C21	0.3219 (7)	0.4647 (13)	0.0398 (3)	0.155 (3)
H21A	0.3817	0.5221	0.0033	0.233*
H21B	0.2204	0.4061	0.0185	0.233*
H21C	0.3896	0.3649	0.0646	0.233*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0597 (15)	0.0751 (19)	0.0595 (15)	0.0205 (16)	0.0030 (13)	−0.0023 (15)
O2	0.0836 (18)	0.089 (2)	0.0568 (16)	0.0033 (17)	0.0014 (14)	0.0036 (16)
O3	0.0504 (14)	0.095 (2)	0.0679 (17)	−0.0066 (15)	0.0228 (13)	−0.0126 (15)

C1	0.055 (2)	0.064 (3)	0.061 (3)	−0.003 (2)	0.007 (2)	−0.004 (2)
C2	0.053 (2)	0.058 (2)	0.058 (2)	0.004 (2)	0.0127 (18)	−0.009 (2)
C3	0.0423 (18)	0.045 (2)	0.055 (2)	−0.0029 (18)	0.0094 (17)	−0.005 (2)
C4	0.0448 (19)	0.058 (2)	0.056 (2)	0.008 (2)	0.0100 (18)	−0.002 (2)
C5	0.059 (2)	0.058 (2)	0.068 (2)	0.014 (2)	0.007 (2)	−0.019 (2)
C6	0.057 (2)	0.056 (2)	0.060 (2)	0.010 (2)	0.0078 (19)	−0.012 (2)
C7	0.0447 (18)	0.040 (2)	0.0503 (19)	0.0036 (17)	0.0103 (17)	0.0011 (17)
C8	0.0440 (18)	0.044 (2)	0.053 (2)	−0.0012 (18)	0.0121 (17)	0.0024 (18)
C9	0.064 (2)	0.040 (2)	0.072 (3)	0.008 (2)	0.005 (2)	−0.001 (2)
C10	0.067 (2)	0.051 (2)	0.062 (2)	0.008 (2)	0.007 (2)	0.003 (2)
C11	0.055 (2)	0.052 (2)	0.049 (2)	0.001 (2)	0.0124 (17)	0.006 (2)
C12	0.061 (2)	0.068 (3)	0.055 (2)	0.001 (2)	0.002 (2)	0.001 (2)
C13	0.064 (2)	0.087 (3)	0.049 (2)	0.005 (3)	0.007 (2)	0.011 (2)
C14	0.062 (3)	0.092 (3)	0.054 (2)	0.003 (3)	0.005 (2)	0.012 (2)
C15	0.077 (3)	0.104 (4)	0.059 (3)	0.017 (3)	0.011 (2)	0.010 (3)
C16	0.077 (3)	0.162 (6)	0.082 (3)	0.027 (4)	−0.002 (3)	0.020 (4)
C17	0.127 (5)	0.234 (9)	0.146 (5)	0.070 (7)	−0.028 (4)	0.012 (7)
C18	0.058 (2)	0.045 (2)	0.066 (2)	−0.0025 (19)	0.010 (2)	0.005 (2)
C19	0.108 (3)	0.092 (4)	0.080 (3)	0.015 (3)	−0.020 (3)	−0.022 (3)
C20	0.180 (6)	0.135 (6)	0.117 (4)	0.070 (5)	0.005 (4)	−0.031 (4)
C21	0.148 (5)	0.170 (7)	0.136 (5)	0.034 (6)	−0.046 (4)	−0.051 (6)

Geometric parameters (Å, °)

O1—C1	1.357 (4)	C11—H11	0.9800
O1—C4	1.464 (4)	C12—C13	1.490 (5)
O2—C1	1.213 (4)	C12—C19	1.520 (5)
O3—C4	1.383 (4)	C12—H12	0.9800
O3—H3	0.8200	C13—C14	1.294 (5)
C1—C2	1.439 (5)	C13—H13	0.9300
C2—C3	1.317 (4)	C14—C15	1.493 (6)
C2—H2	0.9300	C14—H14	0.9300
C3—C8	1.472 (4)	C15—C20	1.534 (7)
C3—C4	1.503 (5)	C15—C16	1.544 (7)
C4—C5	1.507 (5)	C15—H15	0.9800
C5—C6	1.536 (5)	C16—C21	1.512 (8)
C5—H5A	0.9700	C16—C17	1.551 (9)
C5—H5B	0.9700	C16—H16	0.9800
C6—C7	1.528 (4)	C17—H17A	0.9600
C6—H6A	0.9700	C17—H17B	0.9600
C6—H6B	0.9700	C17—H17C	0.9600
C7—C18	1.524 (5)	C18—H18A	0.9600
C7—C11	1.542 (5)	C18—H18B	0.9600
C7—C8	1.565 (5)	C18—H18C	0.9600
C8—C9	1.507 (5)	C19—H19A	0.9600
C8—H8	0.9800	C19—H19B	0.9600
C9—C10	1.536 (4)	C19—H19C	0.9600
C9—H9A	0.9700	C20—H20A	0.9600
C9—H9B	0.9700	C20—H20B	0.9600

C10—C11	1.560 (5)	C20—H20C	0.9600
C10—H10A	0.9700	C21—H21A	0.9600
C10—H10B	0.9700	C21—H21B	0.9600
C11—C12	1.535 (5)	C21—H21C	0.9600
C1—O1—C4	108.5 (3)	C7—C11—H11	107.0
C4—O3—H3	109.5	C10—C11—H11	107.0
O2—C1—O1	120.7 (4)	C13—C12—C19	110.5 (3)
O2—C1—C2	129.5 (4)	C13—C12—C11	110.6 (3)
O1—C1—C2	109.7 (3)	C19—C12—C11	113.0 (3)
C3—C2—C1	108.9 (3)	C13—C12—H12	107.5
C3—C2—H2	125.5	C19—C12—H12	107.5
C1—C2—H2	125.5	C11—C12—H12	107.5
C2—C3—C8	135.5 (3)	C14—C13—C12	127.0 (4)
C2—C3—C4	109.7 (3)	C14—C13—H13	116.5
C8—C3—C4	114.5 (3)	C12—C13—H13	116.5
O3—C4—O1	109.0 (3)	C13—C14—C15	129.3 (4)
O3—C4—C3	113.4 (3)	C13—C14—H14	115.4
O1—C4—C3	103.2 (3)	C15—C14—H14	115.4
O3—C4—C5	108.9 (3)	C14—C15—C20	108.9 (3)
O1—C4—C5	110.6 (3)	C14—C15—C16	110.4 (4)
C3—C4—C5	111.6 (3)	C20—C15—C16	112.9 (4)
C4—C5—C6	111.2 (3)	C14—C15—H15	108.2
C4—C5—H5A	109.4	C20—C15—H15	108.2
C6—C5—H5A	109.4	C16—C15—H15	108.2
C4—C5—H5B	109.4	C21—C16—C15	110.8 (4)
C6—C5—H5B	109.4	C21—C16—C17	110.1 (5)
H5A—C5—H5B	108.0	C15—C16—C17	110.7 (6)
C7—C6—C5	113.2 (3)	C21—C16—H16	108.4
C7—C6—H6A	108.9	C15—C16—H16	108.4
C5—C6—H6A	108.9	C17—C16—H16	108.4
C7—C6—H6B	108.9	C16—C17—H17A	109.5
C5—C6—H6B	108.9	C16—C17—H17B	109.5
H6A—C6—H6B	107.8	H17A—C17—H17B	109.5
C18—C7—C6	110.9 (3)	C16—C17—H17C	109.5
C18—C7—C11	110.2 (3)	H17A—C17—H17C	109.5
C6—C7—C11	117.6 (3)	H17B—C17—H17C	109.5
C18—C7—C8	110.3 (3)	C7—C18—H18A	109.5
C6—C7—C8	107.7 (2)	C7—C18—H18B	109.5
C11—C7—C8	99.3 (3)	H18A—C18—H18B	109.5
C3—C8—C9	121.9 (3)	C7—C18—H18C	109.5
C3—C8—C7	109.5 (3)	H18A—C18—H18C	109.5
C9—C8—C7	104.4 (2)	H18B—C18—H18C	109.5
C3—C8—H8	106.7	C12—C19—H19A	109.5
C9—C8—H8	106.7	C12—C19—H19B	109.5
C7—C8—H8	106.7	H19A—C19—H19B	109.5
C8—C9—C10	103.6 (3)	C12—C19—H19C	109.5
C8—C9—H9A	111.0	H19A—C19—H19C	109.5
C10—C9—H9A	111.0	H19B—C19—H19C	109.5
C8—C9—H9B	111.0	C15—C20—H20A	109.5

C10—C9—H9B	111.0	C15—C20—H20B	109.5
H9A—C9—H9B	109.0	H20A—C20—H20B	109.5
C9—C10—C11	107.3 (3)	C15—C20—H20C	109.5
C9—C10—H10A	110.3	H20A—C20—H20C	109.5
C11—C10—H10A	110.3	H20B—C20—H20C	109.5
C9—C10—H10B	110.3	C16—C21—H21A	109.5
C11—C10—H10B	110.3	C16—C21—H21B	109.5
H10A—C10—H10B	108.5	H21A—C21—H21B	109.5
C12—C11—C7	119.6 (3)	C16—C21—H21C	109.5
C12—C11—C10	112.0 (3)	H21A—C21—H21C	109.5
C7—C11—C10	103.6 (3)	H21B—C21—H21C	109.5
C12—C11—H11	107.0		
C4—O1—C1—O2	177.5 (3)	C11—C7—C8—C3	179.2 (2)
C4—O1—C1—C2	0.3 (4)	C18—C7—C8—C9	−68.6 (3)
O2—C1—C2—C3	−176.2 (4)	C6—C7—C8—C9	170.2 (3)
O1—C1—C2—C3	0.7 (4)	C11—C7—C8—C9	47.1 (3)
C1—C2—C3—C8	172.0 (4)	C3—C8—C9—C10	−160.2 (3)
C1—C2—C3—C4	−1.4 (4)	C7—C8—C9—C10	−35.8 (4)
C1—O1—C4—O3	119.8 (3)	C8—C9—C10—C11	10.5 (4)
C1—O1—C4—C3	−1.1 (4)	C18—C7—C11—C12	−48.5 (4)
C1—O1—C4—C5	−120.5 (3)	C6—C7—C11—C12	79.9 (4)
C2—C3—C4—O3	−116.3 (4)	C8—C7—C11—C12	−164.4 (3)
C8—C3—C4—O3	68.8 (4)	C18—C7—C11—C10	76.9 (3)
C2—C3—C4—O1	1.5 (4)	C6—C7—C11—C10	−154.6 (3)
C8—C3—C4—O1	−173.4 (3)	C8—C7—C11—C10	−38.9 (3)
C2—C3—C4—C5	120.3 (3)	C9—C10—C11—C12	148.9 (3)
C8—C3—C4—C5	−54.7 (4)	C9—C10—C11—C7	18.7 (4)
O3—C4—C5—C6	−76.4 (4)	C7—C11—C12—C13	172.9 (3)
O1—C4—C5—C6	163.7 (3)	C10—C11—C12—C13	51.6 (4)
C3—C4—C5—C6	49.5 (4)	C7—C11—C12—C19	−62.7 (4)
C4—C5—C6—C7	−53.5 (4)	C10—C11—C12—C19	176.0 (3)
C5—C6—C7—C18	−64.2 (4)	C19—C12—C13—C14	121.2 (5)
C5—C6—C7—C11	167.6 (3)	C11—C12—C13—C14	−112.9 (5)
C5—C6—C7—C8	56.6 (4)	C12—C13—C14—C15	176.2 (4)
C2—C3—C8—C9	7.5 (6)	C13—C14—C15—C20	−111.3 (6)
C4—C3—C8—C9	−179.3 (3)	C13—C14—C15—C16	124.2 (5)
C2—C3—C8—C7	−114.6 (5)	C14—C15—C16—C21	−61.1 (6)
C4—C3—C8—C7	58.7 (4)	C20—C15—C16—C21	176.7 (5)
C18—C7—C8—C3	63.4 (3)	C14—C15—C16—C17	176.5 (4)
C6—C7—C8—C3	−57.8 (3)	C20—C15—C16—C17	54.3 (6)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2^{i}	0.82	1.96	2.771 (4)	171

Symmetry codes: (i) −*x*+3, *y*+1/2, −*z*+1.

Fig. 1

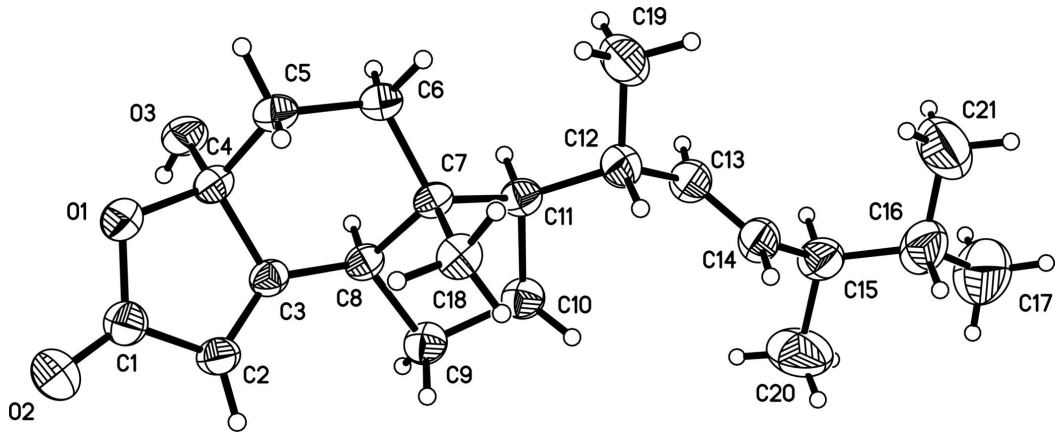
C19
C5
C6
O3
C4
C12
C21
C7
C13
O1
C11
C16
C14
C3
C8
C18
C10
C15
C17
C1
C2
C9
O2
C20

Fig. 2